

**MICROMECHANICAL CHARACTERIZATION AND TEXTURE ANALYSIS OF DIRECT  
CAST TITANIUM ALLOYS STRIPS**

**THE FINAL REPORT**

**For NAG 1 1866 Proposal Funded through LaRC**

**TO:**

**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION**

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# **MICROMECHANICAL CHARACTERIZATION AND TEXTURE ANALYSIS OF DIRECT CAST TITANIUM ALLOYS STRIPS**

## **Summary**

This research was conducted to determine a post-processing technique to optimize mechanical and material properties of a number of Titanium based alloys and aluminides processed via Melt Overflow Solidification Technique (MORST). This technique was developed by NASA for the development of thin sheet titanium and titanium aluminides used in high temperature applications. The materials investigated in this study included conventional titanium alloy strips and foils, Ti-1100, Ti-24Al-11Nb (Alpha-2), and Ti-48Al-2Ta (Gamma). The methodology used included micro-characterization, heat-treatment, mechanical processing and mechanical testing. Characterization techniques included optical, electron microscopy, and x-ray texture analysis. The processing included heat-treatment and mechanical deformation through cold rolling. The initial as-cast materials were evaluated for their microstructure and mechanical properties. Different heat-treatment and rolling steps were chosen to process these materials. The properties were evaluated further and a processing relationship was established in order to obtain an optimum processing condition.

The results showed that the as-cast material exhibited a Widmanstatten (fine grain) microstructure that developed into a microstructure with larger grains through processing steps. The texture intensity showed little change for all processing performed in this investigation. The

heat-treatment followed by cold rolling exhibited a fine-grain microstructure similar to the as-cast material. The overall ductility improved and the ultimate tensile strength decreased. Heat-treating Ti-1100 to 600°C followed by cold rolling can optimize the mechanical and material properties of this material. The heat-treatment of Ti-24Al-11Nb produced a lamellar microstructure with improved ductility. Slight rolling of the material resulted in an improvement of mechanical properties. The results of mechanical anisotropy showed a variation of 30% to 60% in strength over orientations ranging from 0° to 90° for both materials.

The microstructures, crystallographic textures, and mechanical properties have also been investigated in commercial titanium and  $\gamma$ -TiAl alloy sheets strips and foils processed via the melt overflow rapid solidification technology (MORST) technique. The direct cast (DC) foils were fully dense and exhibited equiaxed transformed grain structures and weak  $\{11\bar{2}0\}$ //normal direction solidification textures. After cold rolling, split  $\{0002\}$  textures were observed in both DC and ingot metallurgy (IM) processed foils with the basal poles concentrated approximately 30° from the normal direction towards the transverse direction. Crystallite Orientation Distribution Function analysis indicated the presence of an orientation tube in the cast specimens near  $(\bar{1}01\ 10)[1\bar{2}10]$  and  $(\bar{1}018)[0\bar{1}10]$ . It is suggested that these textures are a result of the lattice rotations and nonuniform cooling that occur during the casting process. After rolling and annealing, main texture orientations of  $(10\bar{1}3)[10\bar{1}1]$  were observed. The mechanical properties of the DC foils were comparable to IM foils. The chill cast ingots exhibited coarse lamellar  $\alpha_2+\gamma$  structures with the lamellae oriented perpendicular to the direction of heat flow. These lamellar arrangements imparted strong  $\langle 111 \rangle_\gamma$  and  $\langle 0001 \rangle_{\alpha_2}$  fiber textures (~10 times random and 19

times random respectively) perpendicular to the chill walls. The results suggest that high quality titanium foils can be processed via MORST without the need for costly and wasteful hot rolling and annealing steps resulting in reduced processing costs.

The result of this project shows that there is an alternative cost effective technique of metal sheet forming process for high temperature alloys of Titanium and Titanium Aluminides which is part of NASA's long term objectives to produce light element alloys for aircraft industries. This will benefit the society by making our aircraft industries more competitive in the world market.

Four undergraduates: Gabrielle Penn, Candice Henderson, Mathew Thames and Lathanza Williams together with one graduate students Enga Wright have been involved in this Project. Three of the undergraduates (Candice Henderson, Mathew Thames and Lathanza Williams) continued to graduate school. Lathanza Williams decided to stay at FAMU working for Dr. Garmestani through a funding by NASA (CENNAS). Dr. Mark Weaver a Post Doctoral Research Scientist funded through a NASA center (Cennas) together with Dr. H. Garmestani have been in charge of this project which resulted in 6 different publications. Dr. Weaver accepted a professorship in University of Alabama in 1998.

The following report contains the detail of the research on four different materials as outlined in four chapters.

# **CHAPTER I**

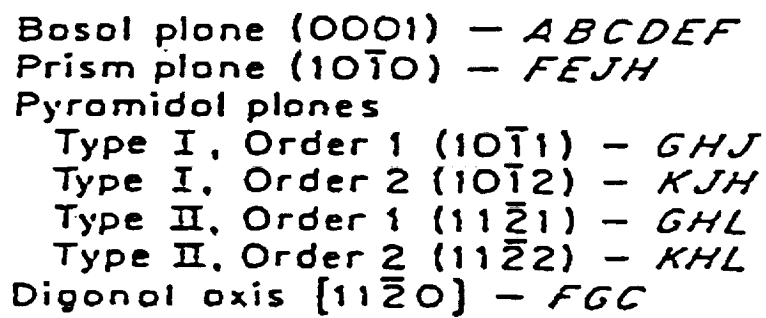
## **MICROCHARATERIZATION OF Ti-1100 PROCESSED via MELT OVERFLOW RAPID SOLIDIFICATION TECHNIQUE**

### **Introduction**

Development of new high temperature materials with optimum properties for high temperature automotive engines, aerospace, composites, superconductors, chemical and petrochemical plants, offshore drilling, and condenser tubing for nuclear plants have been a major focus of research for the past few decades. These materials can be produced at a low cost and high technical benefit, which makes them very attractive for commercial use. There has been increasing interest regarding the evolution of texture and microstructure of these materials [1-4]. The main focus of research in this area seems to be directed at low cost development and fabrication of these high temperature materials with optimum mechanical and material properties. Interest for use in composites is also a reason for interest in titanium and its alloys. Materials alone may exhibit directional dependence (anisotropy) but as a composite it tends to exhibit improved properties.

Ribbon Technology Incorporated, Columbus, Ohio, has developed a method of fabrication for thin sheet titanium foils via Melt Overflow Rapid Solidification Technique (MORST) which eliminates hot working steps and reduces production costs. The problem is the final products in the as-cast condition exhibiting poor properties as a result of the wrinkles produced on the material surface during the MORST process. The fabrication technology by itself does not allow for the production of homogenous thin sheets with optimum properties, as a matter of fact, the sheets are slightly wrinkled and exhibit poor fatigue, material and mechanical properties. This research is focused on determining a post-processing method that produces optimum mechanical and material properties.

Near-alpha alloy, Ti-1100 was used for the present research. A major disadvantage of titanium becomes apparent during conventional production methods of titanium. Production does not always result in the same optimum properties; therefore, it becomes very costly. The result of micro-characterization via crystallographic texture, microstructure analysis (microscopy) and tensile tests are summarized for the post-processing procedures. Microstructure and texture are affected by processing history; therefore, processes have to be chosen carefully. One must understand the structure of the material before proceeding with production. Crystal structure of titanium in its pure condition is hexagonal close packed (hcp) as seen in Figure 1. Titanium generally has a hcp structure but depending on the temperature application of the material. It may also have a body-centered cubic (bcc) structure. The changes in phase and structure can usually be determined by characterizing the material. The objective of this paper is to characterize Ti-1100 based on a few proposed post-processing (heat-treatment and rolling), and interpret what may be done to improve the material after initial processing to ensure optimum properties.



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## **Historical Background**

Titanium was discovered about 1790 as a mineral known as “rutile”. Interest in titanium and its alloys did not begin to escalate until the 1950’s when its principal properties began to be recognized. Titanium was principally produced by the United States, Germany, and Japan in 1955 but now is generally produced in many countries by many companies and industries [2]. Produced in several forms, titanium is available in commercially pure form or may be one of numerous alloys. The alloys include near-alpha, intermetallic, alpha, beta and several other alloys mentioned and explained in depth in the **Materials Properties Handbook: Titanium Alloys**. [1].

Applications for titanium vary and many factors are considered before determining its use. For titanium alloys the size, morphology and distribution of the hcp ( $\alpha$ -phase) and bcc ( $\beta$ -phase) have a strong influence on the mechanical properties [3]. Properties such as high strength-to-weight ratio, corrosion resistance, fatigue strength, and creep resistance are the reasons that titanium is considered for high temperature applications [1, 4]. Near-alpha alloys are among those systems receiving some attention for development of advanced materials for high temperature applications in aerospace, automotive and other industries. The physical properties of Ti-1100 are summarized in Table 1 [1]. The phase diagram (Figure 2) shows the phases for various weight compositions of aluminum in titanium.



TABLE 1: Mechanical and Material Properties of Ti-1100.

Property	Ti-1100
Beta transus (nominal)	1015°C (1860°F)
Density, g/cm <sup>3</sup> (lb/in <sup>3</sup> )	4.5 (0.163)
Modulus of Elasticity, GPa (10 <sup>6</sup> psi)	107 to 117 (15.5 to 17)
Coefficient of Linear expansion	8.5 x 10 <sup>-6</sup> / °C (4.7 x 10 <sup>-6</sup> / °F)

Source: Tom O'Connell, TIMET in Materials Properties Handbook of Titanium Alloys, 1994, pp. 412

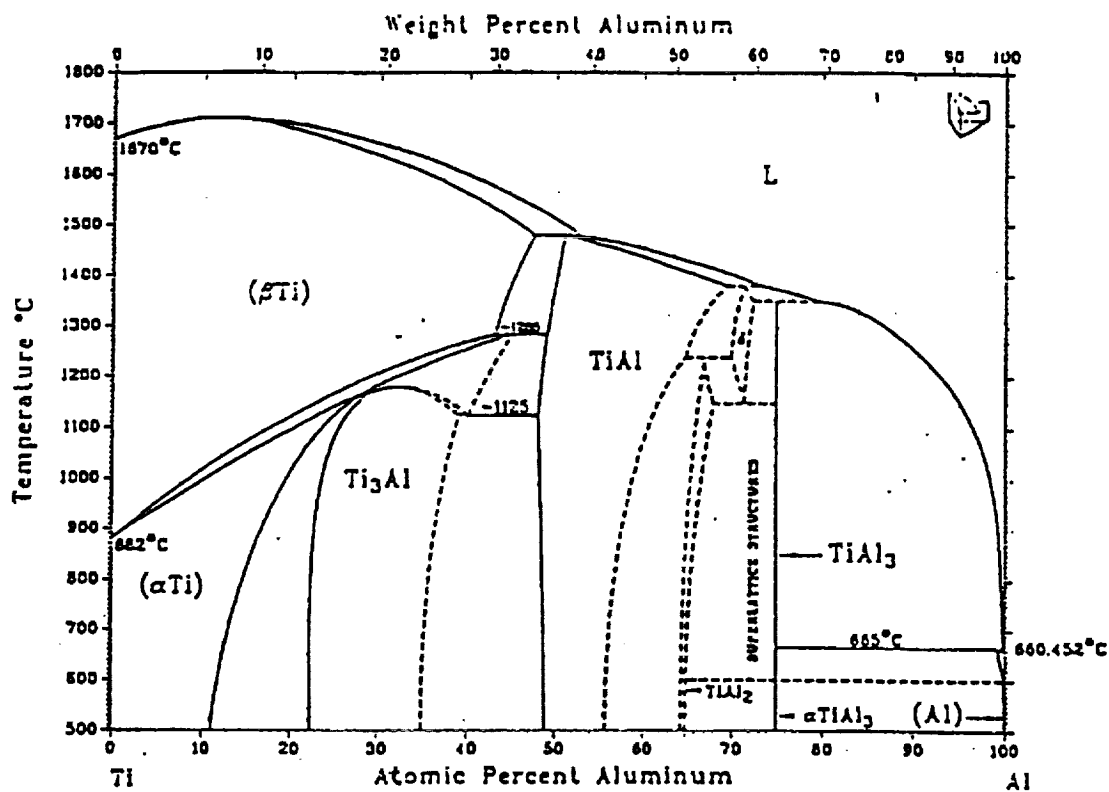


FIGURE 2: Ti-AL BINARY PHASE DIAGRAM.

## **Near-Alpha Titanium Alloys**

Ti-1100 is a silicon-bearing near-alpha titanium alloy developed by TIMET Corporation [5]. It has the composition Ti-6Al-2.75Sn-4Zr-0.4Mo-0.45Si-0.07O<sub>2</sub>-0.02Fe at its maximum. The exact composition of the Ti-1100 material used for this research was not available. It was developed for elevated-temperature use up to 600°C (1100°F). It was designed for applications requiring excellent creep strength or fracture properties at high temperatures such as, high-pressure compressor disks, low-pressure turbine blades, and automotive valves [1]. Ti-1100 offers the highest combination of strength, creep resistance, fracture toughness and stability of any commercially available titanium alloys [1]. Table 2 shows the typical composition range of Ti-1100. Used primarily in the beta-processed condition, the typical microstructure for Ti-1100 is equiaxed  $\alpha$ - $\beta$  for billet and sheet rock. The cooling rate, which affected the initial samples prepared for this research, transforms the  $\beta$  structure to a Widmanstatten or colony  $\alpha + \beta$  structure [1]. Usually a smaller grain size exhibits better ductility but, TIMET suggests a lamellar microstructure, which consists of large transformed  $\beta$ -grains due to annealing above the  $\beta$ -transus temperature ( $T_{\beta} = 1010\text{C}$ ), exhibits better creep and fatigue crack growth properties [3]. Therefore, it becomes hard to optimize all properties within a single microstructure. All of the factors outlined above were taken into consideration during testing and analysis.

TABLE 2: Ti-1100 Typical Composition Range.

	Composition, wt. %								
	Al	Sn	Zr	Fe	Mo	Si	O <sub>2</sub>	N <sub>2</sub>	C
Minimum	5.7	2.4	3.5	...	0.35	0.35	...	...	...
Maximum	6.3	3.0	4.5	0.02	0.50	0.50	0.09	0.03	0.04
Nominal	6.0	2.7	4.0	...	0.40	0.45	0.07	...	...

Source: Tom O'Connell, TIMET in Materials Properties Handbook of Titanium Alloys, 1994

## Fabrication/ Processing

Fabrication, or production, of titanium and its alloys were first demonstrated in the United States in 1954 using machined high-density graphite molds at the U.S. Bureau of Mines. Since the titanium casting industry is relatively young the high reactivity of titanium presents a challenge to the foundry. Special methods are required to maintain metal integrity. Titanium alloys are produced by all forging methods currently available. Selection of the optimal forging is based on the desired shape, the cost and desired mechanical properties and microstructure. There are several ways to reach a desired result and it differs from material to material. After initial processing, some post-processing may be required to achieve optimum results. There are several post-processing methods, such as heat treatment and hot or cold rolling. Certain properties of titanium differ between the various alloys. For example, deformation resistance depends on amount and type of the alloying element. In the following, a review of Ti-1100 is provided for their past processing history and is used for comparison later in this work.

## **Ti-1100**

Near- $\alpha$  titanium alloys are not ordinarily in a state of true equilibrium after initial processing, because of the relatively fast rates of cooling from high forging and solution anneal temperatures [8]. Several methods have shown success in eliminating this processing problem. It has been processed to billet, bar, sheet, and weld wire. Ti-1100 may also be hammered or press forged using isothermal, warm die, or conventional die [2]. Ti-1100 has a low tolerance for cold formability. It is sensitive to formation during re-heating processes which may lead to undue surface cracking. Stabilization treatments are generally in the range of 500 to 600°C (930 to 1200°F).

This alloy has been successfully melted for several years. Ti-1100 has a slight response to cooling rate or sample size from the solution treatment (or processing) temperature. On the other hand, very rapid quenching increases strength and decreases creep resistance at elevated temperatures. Ti-1100 comes in two standard alloying forms. One alloy is beta processed and the other is alpha-beta processed. The alpha-beta condition consists of two annealing steps. The material is first beta annealed at  $T > 1065^{\circ}\text{C}$ , ( $1950^{\circ}\text{F}$ ) and then annealed at  $T = 595^{\circ}\text{C}$ , ( $1100^{\circ}\text{F}$ ). Ti-1100 is provided in the equiaxed alpha-beta condition for forming products to enhance the ease of processing. By fabricating this material via MORST it is desired that the disadvantages (extreme cost, optimal properties, hot working steps, etc.) associated with producing Ti-1100 by conventional methods is bypassed. Ribbon Technology (Ribtech) developed a methodology to produce titanium foil sheets via MORST, eliminating excessive hot working steps while keeping the mechanical properties relatively constant.

## Rolling

The majority of specifications for near- $\alpha$  and  $\alpha + \beta$  titanium alloys mandate final hot working in the  $\alpha + \beta$  region ( $T_{\beta} = 1015^{\circ}\text{C}$ ) [2]. Rolling in the  $\alpha + \beta$  region produces microstructures with ductility and low cycle fatigue properties substantially higher than microstructures produced by rolling above the  $\beta$  transus temperature. For this process, rolling temperatures are typically selected at 30 to 55°C (50 to 100°F) below the  $\beta$  transus temperature. This allows for inaccuracies in the  $\beta$  transus temperature determination. Most of the literature in this area focused its attention on hot rolling and very little on cold rolling of titanium alloys. The main reason for this hot rolling process is the lack of ductility at room temperature. The present work uses more cold working rather than hot working to attempt to avoid the problems that occur during heat-treatment of titanium.

## Heat Treatment

The purpose of heat-treating titanium alloys is in order to:

1. Reduce residual stresses developed during fabrication.
2. Produce an optimum combination of ductility, machinability, and dimensional and structural stability (annealing).
3. Optimize special properties such as fracture toughness, fatigue strength, and high-temperature creep strength through creating a lamellar  $\beta$  structure with grain size of interest.
4. To increase ductility and prepare the material for further rolling for the purpose of optimizing the properties.

Several key considerations are made when heat treating titanium alloys. The response of titanium to heat treatment depends on the composition of the metal and the effects of alloying elements on the  $\alpha$ - $\beta$  crystal transformation. For example, near- $\alpha$  alloys can be annealed and stress relieved yet no type of heat treatment will increase its strength unless it involves aging or is followed by rolling or other forming process [2]. When heat-treating titanium it must be cleaned and dried to prevent contamination (or embrittlement) to the material. It must not be cleaned with ordinary tap water. An unclean specimen is subject to stress corrosion.

## Forging

All types of forging are used for titanium and its alloys. They include open-die, closed-die, upsetting, roll, orbital, spin, mandrel, ring and forward forging. Titanium alloys generally

are more difficult to forge than other materials because their deformation resistance can increase dramatically with small changes in metal temperatures and strain rates. Because of this conventional forging is heated to facilitate the forging process and to reduce metal temperature losses which, may lead to excessive cracking. Conventional forging is a term that describes a forging process that is done below the  $\beta$  transus temperature of the alloy [2]. See Table 3 for a comparison of processing capability for Ti-1100 and conventional titanium.

TABLE 3: Processing Capability.

Alloy	Ingot metallurgy	Forging	Sheet rolling	Casting
Ti	Yes	Yes	Yes	Yes
Ti-1100	Yes	Yes	Yes	With difficulty

## **Experimental Methodology**

### **Sample Preparation**

The as-cast Ti-1100 material was fabricated at Ribtech via Melt Overflow Rapid Solidification Technique (MORST). This process involves direct strip casting and cold rolling. Hot-working steps are eliminated. The material was cast in a plasma melt overflow furnace. The furnace combines plasma arc melting in a cold copper hearth with MORST by rotating the cold copper hearth about the same axis of rotation as the chill roll to overflow liquid on to the circumference of the chill roll, see Figure 3 [6].

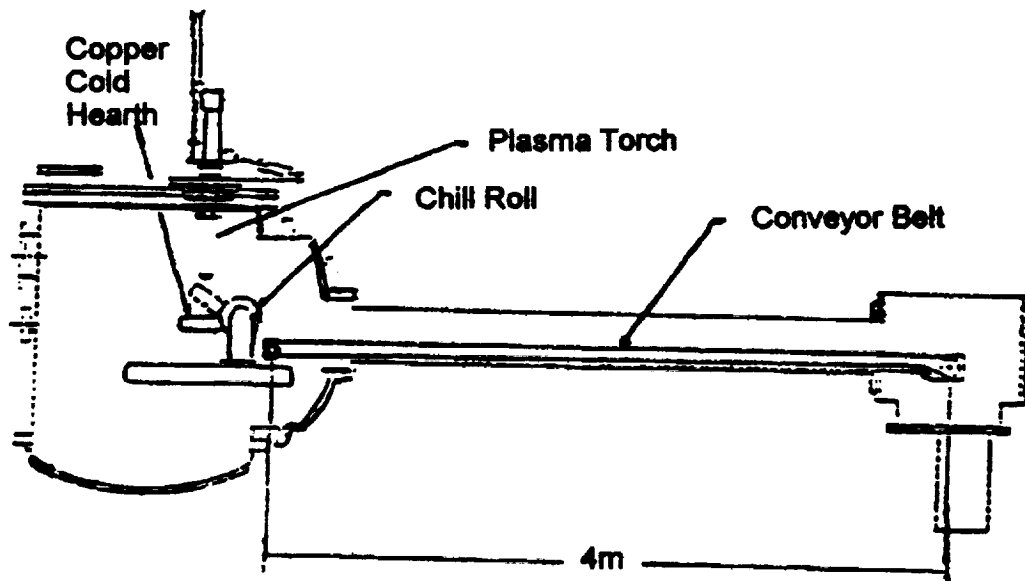
Samples of the material were machined into dog-bone shaped tensile specimens in 0, 30, 45, 60 and 90°C (see Figure 4). Small rectangular squares were cut for textural and microstructural analysis.

### **Heat Treatment**

Different heat treatment procedures were performed to determine the best process. Figure 3 and 4 show the organizational schedule for the heat treatment experiments. The initial texture and microstructure analysis of Ti-1100 and  $\alpha$ -2 was performed using X-ray and ESEM.

The as-cast Ti-1100 material was vacuum annealed at 550°C and 600°C for three hours in an atmosphere of argon gas. The samples were cold rolled in the rolling direction to an optimum strain to determine a suitable post-processing method. The texture, microstructure, and tensile properties of these samples were analyzed and the results are discussed in the next chapter. The heat-treatment temperatures were chosen to determine if the properties could be optimized for the service temperature of the material.





**Solidified Ribbon**

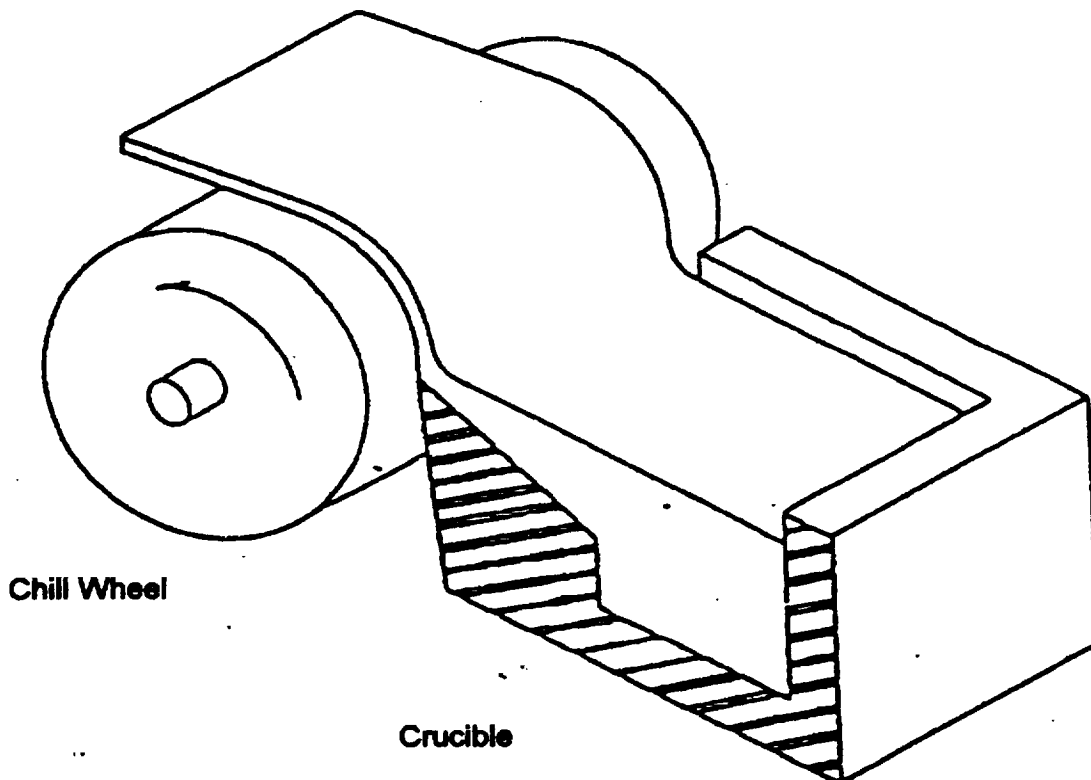


FIGURE 3: DIAGRAM OF THE MORST PROCESS

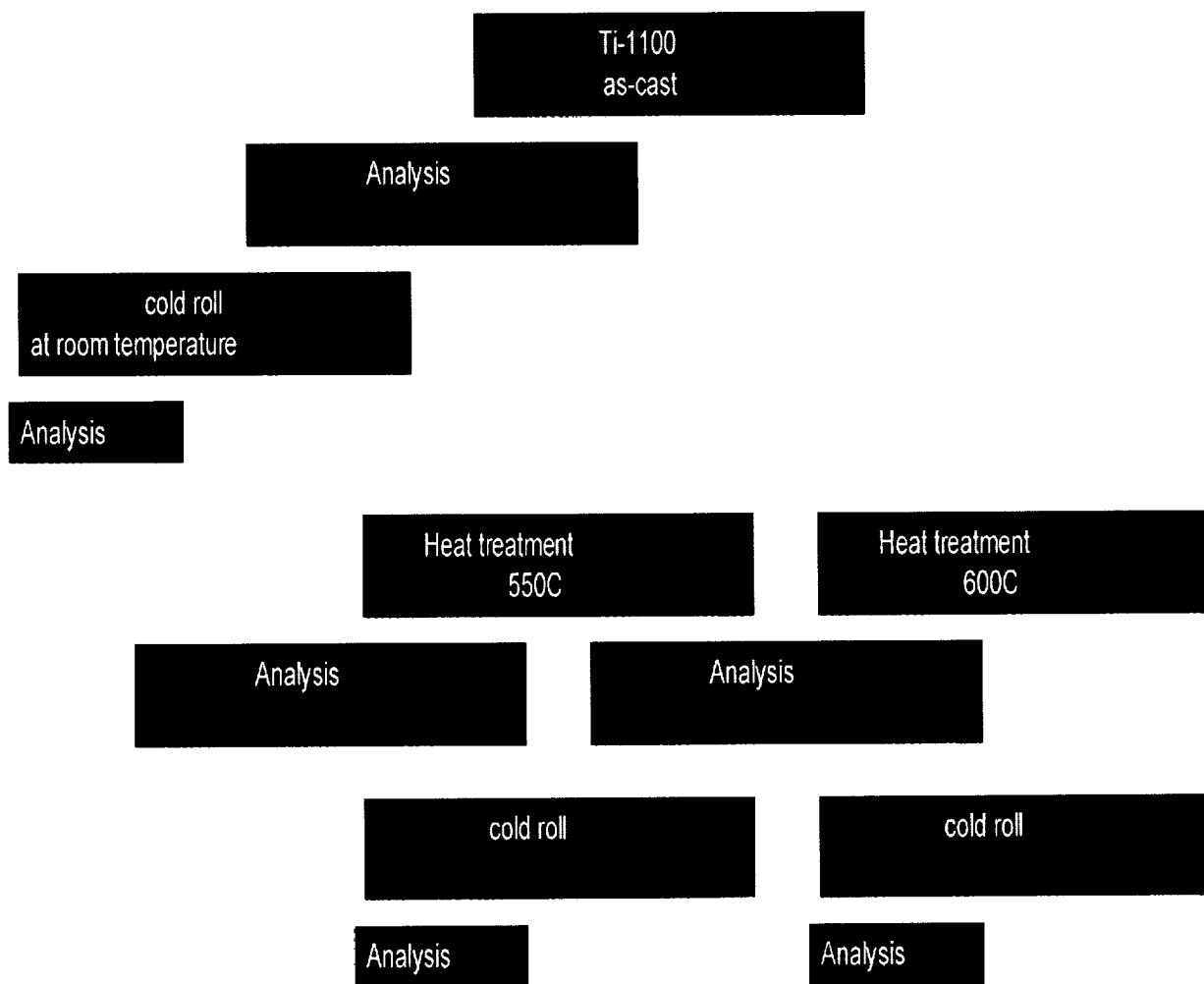


FIGURE 4: POST-PROCESSING METHODS FOR Ti-1100.

The square samples were mounted following heat treatment for ESEM and X-ray analysis. Each sample was polished using a three step polishing procedure (MD-Piano, MD-Plan, and MD-Chem) developed by Struers for titanium metals to produce a flat & stress free surface ideal for texture and micro-structural analysis. The samples were etched prior to microscopy analysis using Kroll's Reagent solution (1-3mL HF, 2-6mL HNO<sub>3</sub>, and 100mL H<sub>2</sub>O). The microstructure was examined using an optical microscope but, the available magnification was found too small for the investigation. Microscopy analysis was then performed using an Environmental Scanning Electron Microscope (ESEM). The ESEM was utilized to further examine the finer microstructure and to perform Electron Dispersive Spectroscopy (EDS). EDS determines the chemical composition differences of the material after processing, which helps to analyze the final results.

Texture was analyzed via the X-ray Diffractometer for samples at all stages of processing. Using Poppa, orientation distribution functions (ODF's) were calculated by Roe's approach to spherical harmonic method. The pole figures were recalculated using the ODF's. The crystallographic planes used to calculate the pole figures for Ti-1100 were as follows: {002}, {100}, {101}, {102} and {110}. Only the  $\phi = 0^\circ, 20^\circ, 30^\circ, 40^\circ$  angles were used from the pole Figure calculations for texture analysis.

## Tensile Test

Tensile specimens were machined with dog-bone shape. These specimens were tested using a servo-hydraulic Mechanical Testing System (MTS). Tests were done on specimens cut in five different orientations ( $0^\circ$ ,  $30^\circ$ ,  $45^\circ$ ,  $60^\circ$  and  $90^\circ$ ) from the rolling direction and the data was compared.

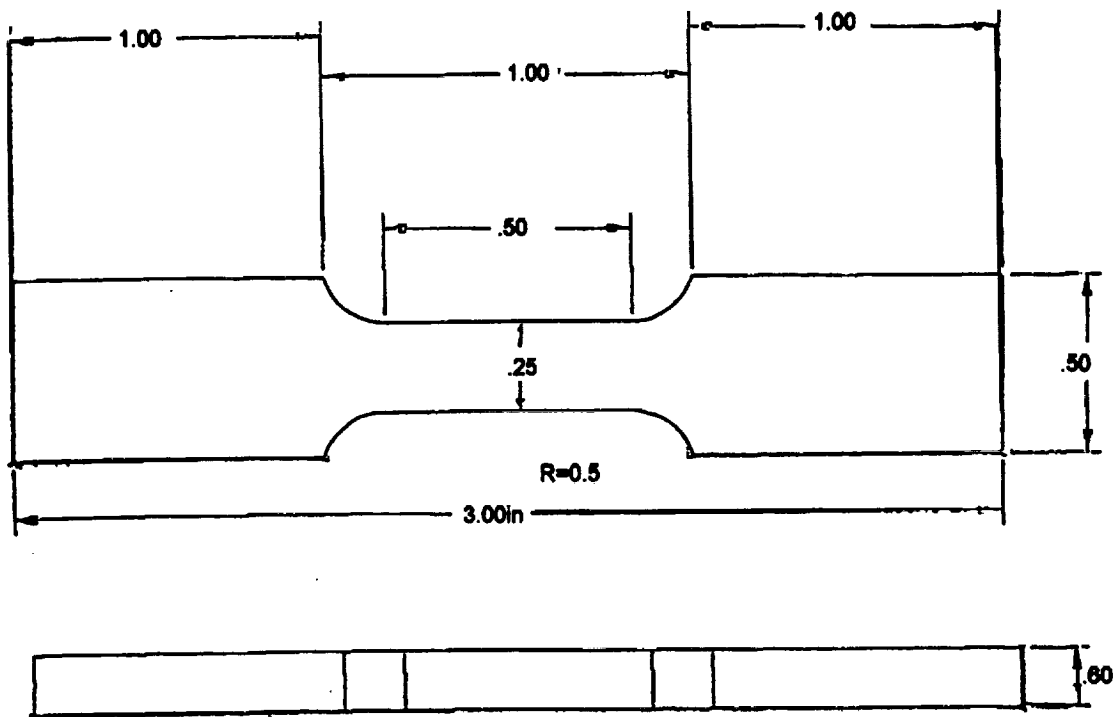


FIGURE 5: DIMENSIONS FOR TENSILE SAMPLES.

## Results and Discussion

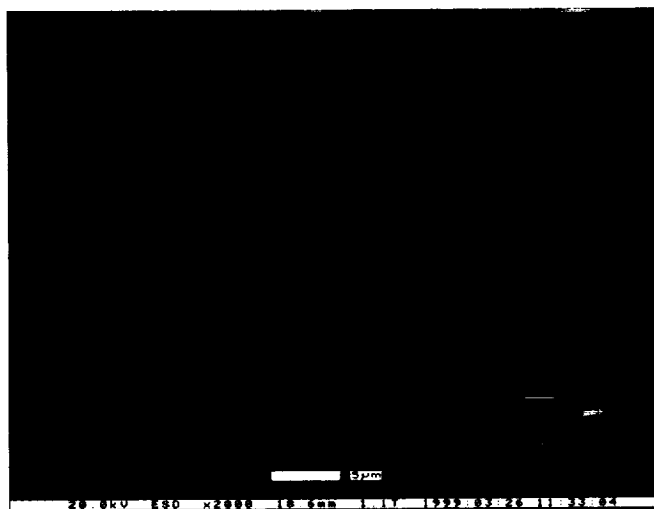
### Microstructure: Ti-1100

The microstructure of the as-cast material was compared to the results from previous studies (3,8). Initial microstructure of the as-cast material was very faint. The material was etched with Kroll's Reagent several times until the microstructure was visible by the ESEM. Once the microstructure was uncovered it exhibited a Widmanstatten structure (basketweave). The rapid cooling rate did not allow the lamellar width to increase during initial processing. Titanium when heat-treated should blue when cooled. It was expected that heat-treatment at 600°C would give similar results as the sample heat-treated at 550°C. Heat-treatment at 550°C, resulted in samples that were blue, but not all the samples heat-treated at 600°C were blue. Although, all were heat-treated in the same atmosphere of argon gas some samples exhibited slight surface oxidation. Other than the discoloration after heat-treatment the results at 550°C and 600°C proved to be similar. Aside from the apparent oxidation the material still exhibited a Widmanstatten microstructure. Since the Ti-1100 material was slow furnace cooled it progressed from the fine-grain Widmanstatten microstructure of the as-cast material to a more coarse-grained microstructure. The results are shown in FIGURE 7, and 8. The samples were then rolled using a manual-rolling machine. The percentage of reduction was determined from the following equation:

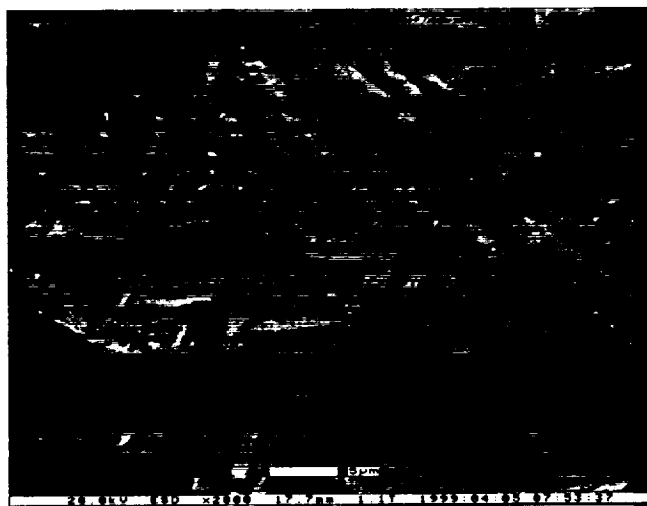
$$\%R = [(T_{avg}^i - T_{avg}^f) / T_{avg}^i] \times 100$$

where %R is the percent reduction,  $T_{avg}^i$  is the initial thickness, and  $T_{avg}^f$  is the final thickness..

The results are presented in Table 5. There was no indication of brittleness.

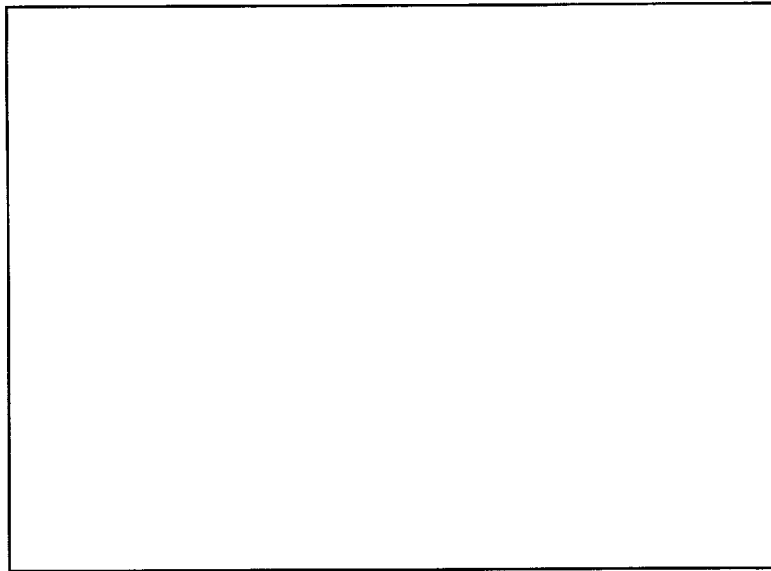


(a)



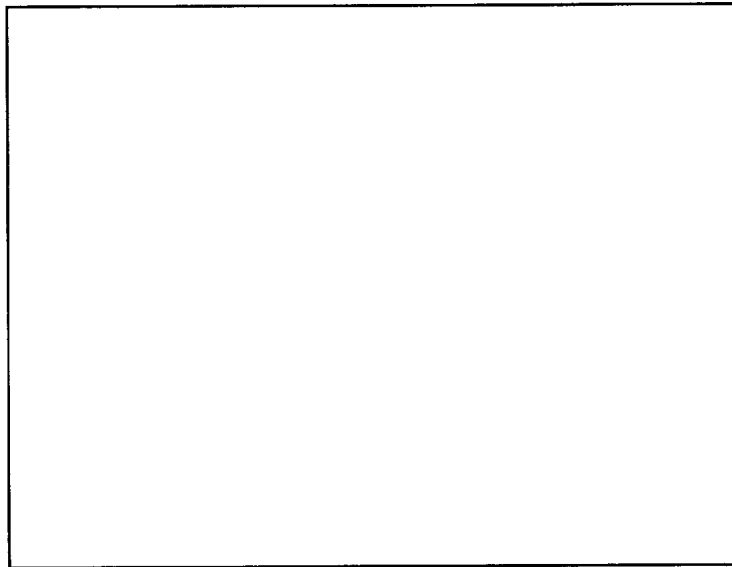
(b)

FIGURE 7: (A) AS-CAST Ti-1100 (B) AS-CAST Ti-1100 ROLLED.



(a)

Figure 8: (a) Annealed @ 550°C for 3h.



(b)

FIGURE 9: ANNEALED @ 550°C FOR 3H AND COLD ROLLED.

## Texture: Ti-1100

Preliminary texture analysis revealed random texture in the as-cast material. The texture analysis process included rolling the as-cast material to determine the outcome of the material before any post-processing. Cold rolling the as-cast material exhibited no change in structure. Figure 10 shows that both as-cast and as-cast cold rolled have the same texture intensity.

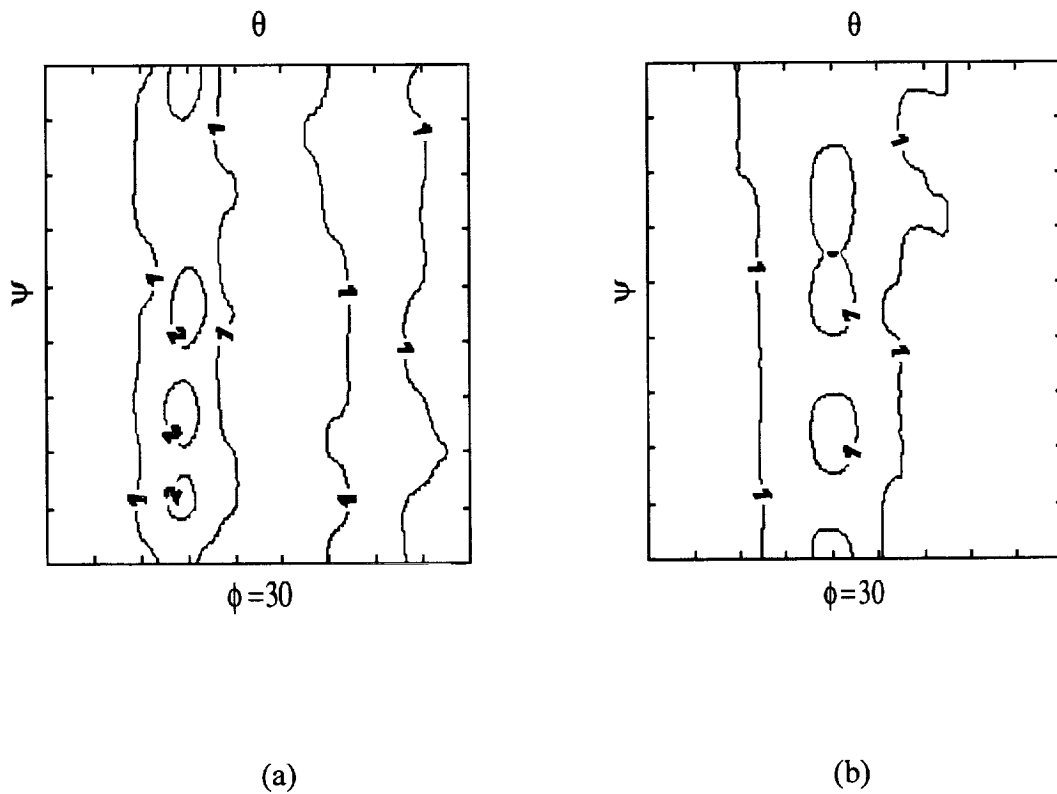


FIGURE 10: (A)Ti-1100 AS-CAST (B) Ti-1100 AS-CAST AND COLD ROLLED..

To the naked eye the Ti-1100 samples that were heat-treated at 550°C were blue in color which means the heat-treatment went well but the texture results show little change. The intensity only increased by one. The samples heat-treated at 600°C had more gold to its color and it exhibited



almost no texture. The highest intensity for any of the samples was 2x random. Ti-1100 was developed for high temperature applications up to 600°C. The results of the post-processed material are shown in Figures 11 and 12.

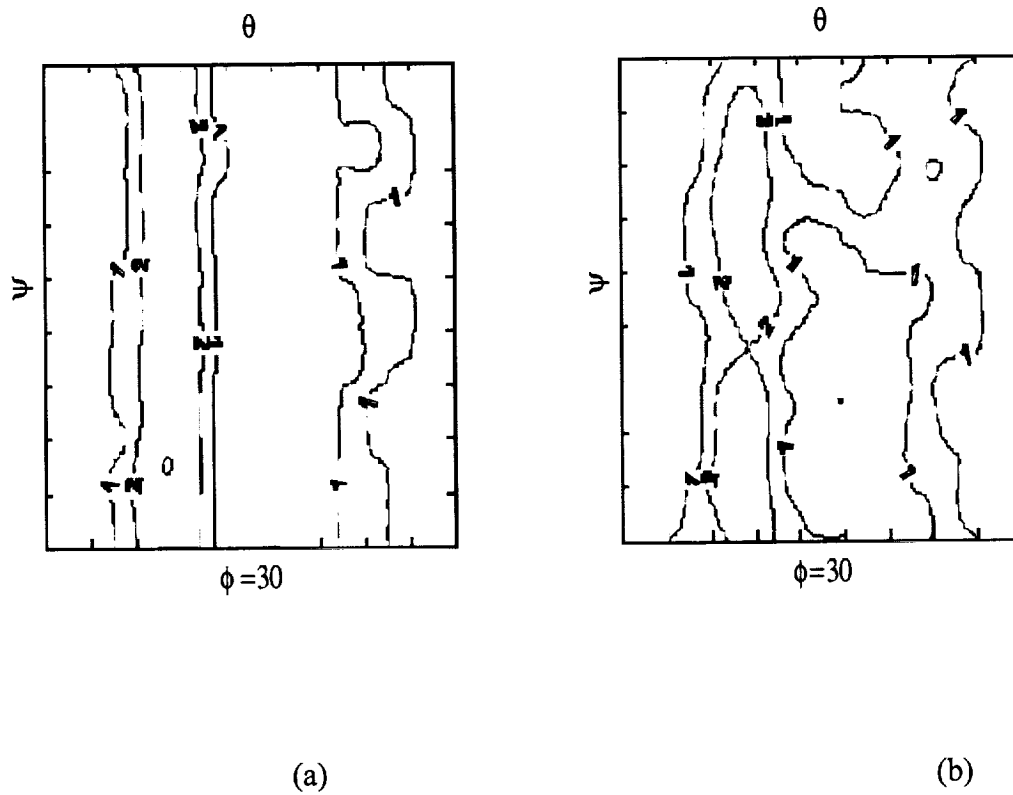
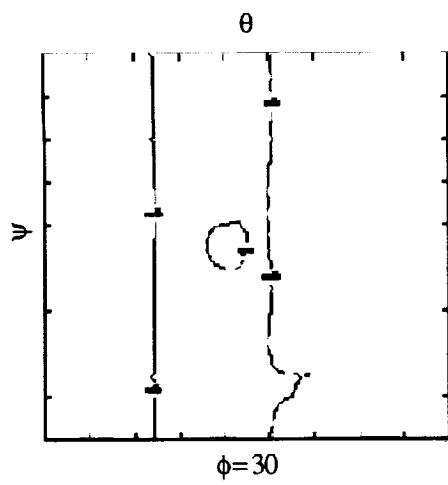
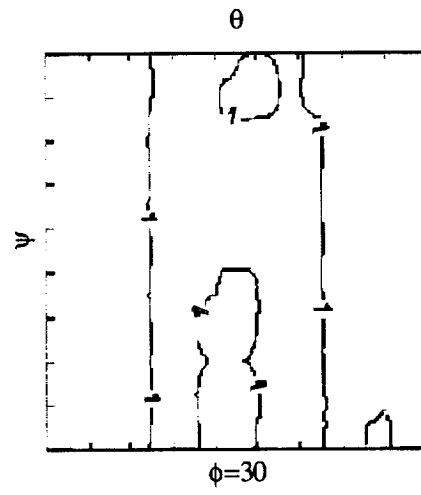


FIGURE 11: (A) Ti-1100 ANNEALED @ 550°C FOR 3H (B) Ti-1100 ANNEALED @ 550°C FOR 3H AND COLD- ROLLED.



(a)



(b)

FIGURE 12: (A) Ti-1100 ANNEALED @ 600°C FOR 3H (B) Ti-1100 ANNEALED @ 600°C FOR 3H AND COLD ROLLED.

TABLE 4: Summary of characterization of Ti-1100.

(times random)

	AS-CAST	HEAT TREATMENT		ROLLED		
		550°C	600°C	AC	550°C	600°C
Microstructure	Widmanstatten	Coarse grains	Coarse grains	Fine grains	Elongated grains	Elongated grains
(2 0 2 1) $\phi = 0^\circ$	2	2	2	1	1	1
(3 0 3 1) $\phi = 0^\circ$	2	2	2	0	1	1
(-3 1 2 1) $\phi = 20^\circ$	2	2	2	1	1	1
(-2 1 1 1) $\phi = 30^\circ$	1	3	3	0	0	1
(-3 2 1 1) $\phi = 40^\circ$	2	2	2	1	1	1
Reduction				2.7%	2.5%	8.4%

## **Mechanical Testing Ti -1100**

Several tensile experiments were performed on Ti-1100 for five orientations ( $0^\circ$ ,  $30^\circ$ ,  $45^\circ$ ,  $60^\circ$ , and  $90^\circ$ ). The experiments were done on both as-cast and heat-treated materials to determine its anisotropic behavior. The limited thickness of the thin sheet material made it difficult to get good readings because of slipping. Data was collected for each experiment. It was averaged to get results. These results were compared. The experiments showed that the ultimate tensile strength (UTS) ranged from 14,500 lbf/in<sup>2</sup> to 91,500 lbf/in<sup>2</sup> for the Ti-1100 material. There were no significant changes present across the orientations. Appendix B shows a graphed summary of the UTS results.

Ductility was highest for the as cast material in the  $0^\circ$  orientation at approximately 13%. On the lower end of ductility .2%, was the  $0^\circ$  orientation of the specimen processed at  $600^\circ\text{C}$ . Observation of the as cast and  $550^\circ\text{C}$  samples showed  $0^\circ$  and  $45^\circ$  with breaks near the center of the specimen but actual results showed a decline in ductility as the orientation increased (2% to 13%). The samples processed at  $600^\circ\text{C}$  was the only one to show an increase in ductility over the orientations (.2% to 8%). The results are shown in Appendix B. Heat-treatment effects change yield stress very little but, ductility changes are found to be more pronounced [5]. This is seen in the tensile test results of the heat-treated samples. Table 5 summarizes the MTS results for all orientations and samples.

TABLE 5: Summary of MTS Results.

<b>SUMMARY OF MTS RESULTS</b>				
Sample	HT	Orientation (deg from RD)	UTS (KSI)	Strain
as-cast Ti-1100	n/a	0	76	0.129
as-cast Ti-1100	n/a	30	91	0.097
as-cast Ti-1100	n/a	45	48	0.059
as-cast Ti-1100	n/a	60	22	0.060
as-cast Ti-1100	n/a	90	20	0.041
Ti-1100	550	0	52	0.059
Ti-1100	550	30	72	0.055
Ti-1100	550	45	32	0.024
Ti-1100	550	60	29	0.0377
Ti-1100	550	90	14	0.0262
Ti-1100	600	0	56	0.0282
Ti-1100	600	30	23	0.0309
Ti-1100	600	45	25	0.0366
Ti-1100	600	60	26	0.0227
Ti-1100	600	90	73	0.0793

## Summary

In summary, a post-processing technique is proposed to optimize the mechanical property and microstructure of the as-cast titanium and titanium aluminide thin sheet material produced by MORST technique. The post processing included several steps of heat-treatment and cold rolling of titanium. The as-cast Ti-1100 exhibited a Widmanstätten microstructure and texture of two times random. This is a result of the fast quenching process of MORST. The initial mechanical characterization shows that the MORST process produces higher strength properties for Ti-1100 materials and a lower strength for  $\alpha$ -2 compared to conventional techniques. The strength of Ti-1100 decreased slightly after the post-processing procedures (heat-treatment and cold rolling). Ti-1100 heat-treated at 600°C exhibited an increasing ultimate tensile strength over the orientations but, still lower than the UTS of the as-cast material. The as-cast material exhibited higher strength and ductility over the processed materials. Further experiments should be done to determine optimal properties above its theoretical working temperature (600°C). In conclusion, to optimize material and mechanical properties of Ti-1100 it should be annealed at 600°C and cold rolled.

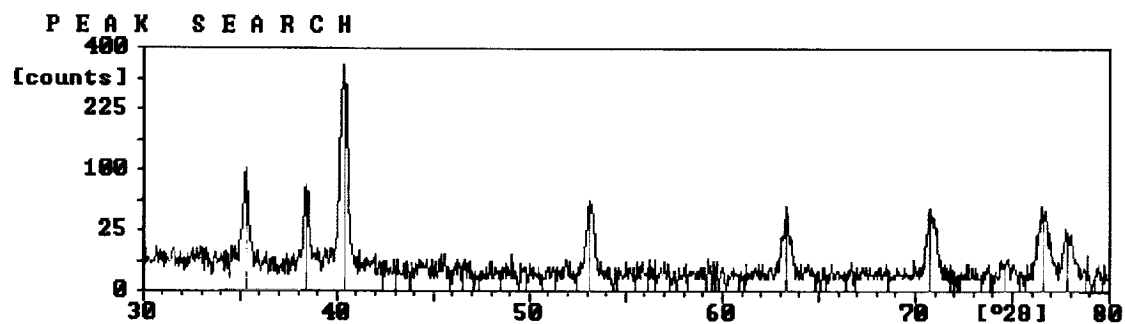
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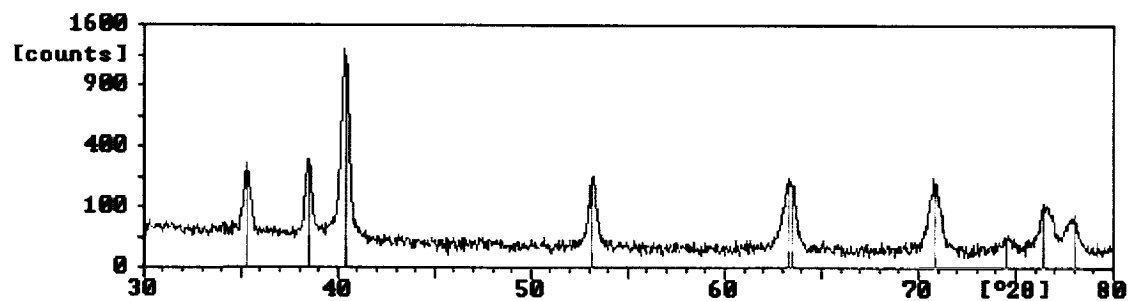
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## APPENDIX A:



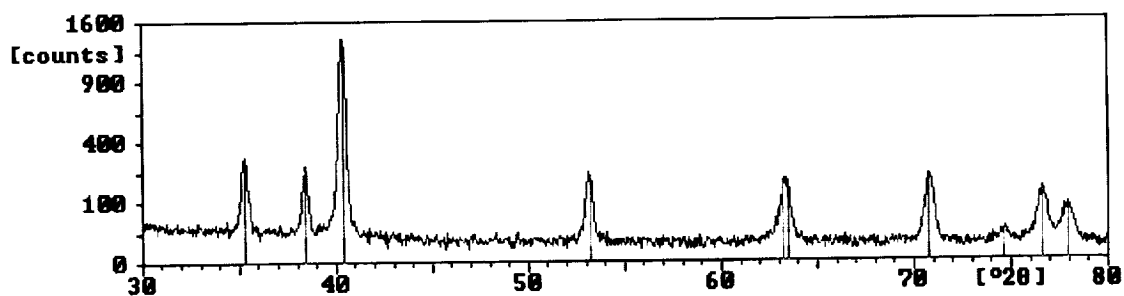
2-Theta Scan for Ti-1100 As-cast



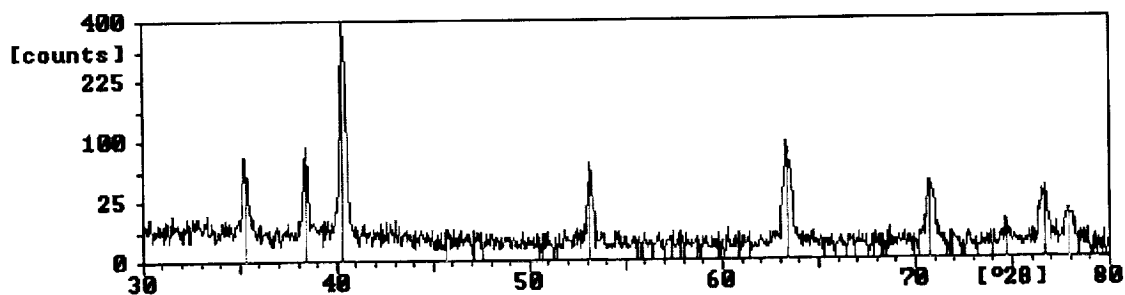
2-Theta Scan for Ti-1100 As cast and cold rolled



2-Theta Scan for Ti-1100 Heat-treated at 550°C for 3h



2-Theta Scan for Ti-1100 Heat-treated at 550°C for 3h and cold rolled

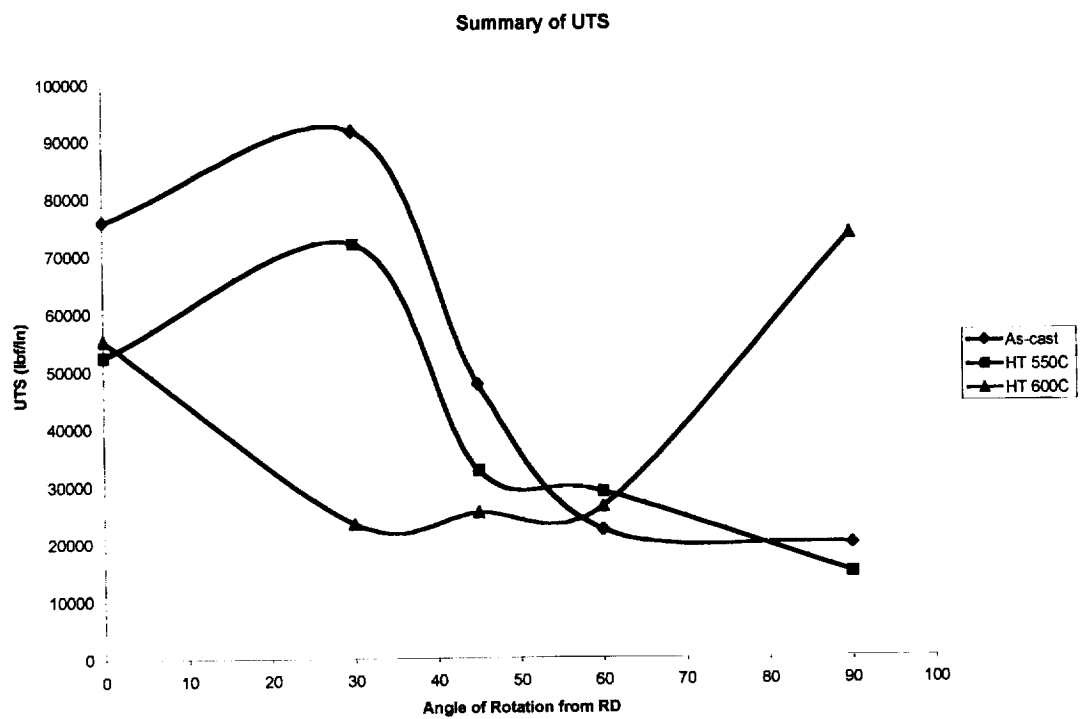


2-Theta Scan for Ti-1100 Heat-treated at 600°C for 3h

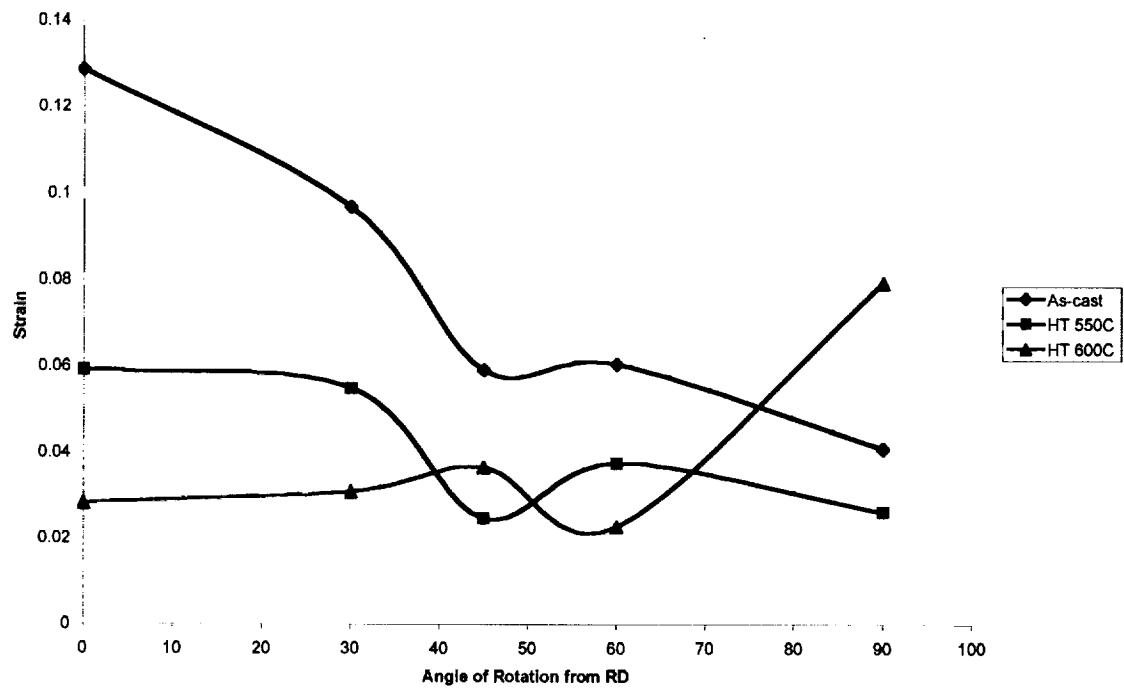


2 -Theta Scan for Ti-1100 Heat-treated at 600°C for 3h and cold rolled

## APPENDIX B



### Summary of Ductility



## CHAPTER II

### TEXTURE STUDIES OF GAMMA TITANIUM ALUMINIDE SHEETS PRODUCED BY MELT OVERFLOW RAPID SOLIDIFICATION

#### INTRODUCTION

A texture analyses have been conducted on gamma titanium aluminide ( $\gamma$ -TiAl) strips produced using the recently developed plasma melt overflow process. The results indicated that  $\langle 101 \rangle$  deformation textures persisted in the  $\gamma$ -phase while  $\langle 0002 \rangle$ ,  $\langle 10\bar{1}0 \rangle$ , and  $\langle 11\bar{2}0 \rangle$  texture components were all observed in the  $\alpha_2$ -phase. After annealing at 1065°C/48 hrs., the  $\gamma$ -phase textures did not change while the  $\alpha_2$ -phase changed from a basal to a  $\langle 10\bar{1}0 \rangle$  texture. It is suggested that the texture development in direct cast  $\gamma$  strips produced using this technique are a direct result of lattice rotations and residual stresses caused by nonuniform cooling.

Gamma titanium aluminides are considered good candidates for use in high temperature structural applications. Their favorable combinations of low density, high elastic modulus, good high-temperature strength retention, and good oxidation resistance allow them to exceed the operating temperatures of advanced titanium alloys and nickel- or iron-based superalloys up to 1073 K [1]. However, like most intermetallic compounds, the use of  $\gamma$ -based alloys has been hindered to some extent by low ductility and fracture toughness at room temperature and to a larger extent by poor formability leading to higher fabrication costs. To overcome some of these obstacles, several research efforts have been directed towards the development of more effective processing techniques and understanding the relationships between processing technique, microstructure and mechanical behavior [2-11]. Recently, Gaspar *et al.* [12-14] have reported success in direct casting of sheets of conventional titanium alloys and ordered intermetallic alloys

using a single-chill-roll casting technique called melt overflow rapid solidification technology (MORST). For example, orthorhombic (Ti-22Al-23Nb) and gamma (Ti-45Al-2Cr-2Nb) based alloys were continuously cast using this process into  $\sim 500 \mu\text{m}$  thick  $\times 10 \text{ cm}$  wide  $\times 3 \text{ m}$  long sheets which were successfully ground, cold rolled, or hot pack rolled to foil gauge ( $\leq 100 \mu\text{m}$  thickness). As with all processing techniques, the method of fabrication and subsequent thermomechanical processes can result in pronounced plastic anisotropy which can greatly influence the resulting mechanical properties. In the present document, the microstructures and textures have been evaluated in direct cast (DC)  $\gamma$  sheets using x-ray diffractometry (XRD) and compared with results for materials produced using conventional ingot metallurgy (IM) processes.

## EXPERIMENTAL PROCEDURE

Gamma titanium aluminide strips were cast in the plasma melt overflow furnace at Ribbon Technology Incorporated, Columbus, Ohio. This furnace combines plasma arc melting in a cold copper hearth with MORST by rotating the cold copper hearth about the same axis of rotation as the chill roll to overflow liquid onto the circumference of the chill roll [12-14]. The analyzed composition was Ti-45.0 at.% Al-2.0 at. % Nb-2.0 at.% Cr, and, in wppm, 100 C, 562 O, 26 H and 83 N. Portions of each alloy strip/sheet were cut into small pieces, mechanically polished, and etched prior to data collection with a solution consisting of 10 ml hydrofluoric acid, 5 ml nitric acid, 35 ml hydrogen peroxide, and 100 ml water. Texture variations were measured using the x-ray diffraction technique on a Philips X'Pert PW 3040 MRD x-ray diffractometer operating at 40 kV and 45 mA. The following incomplete pole figures were measured using Ni filtered  $\text{CuK}_\alpha$  radiation to determine textures in the  $\gamma$  and  $\alpha_2$  phases:  $\{100\}$ ,  $\{111\}$ ,  $\{200\}+\{002\}$ ,  $\{220\} + \{202\}$ ,  $\{0002\}_{\alpha_2}$ ,  $\{20\bar{2}0\}_{\alpha_2}$ ,  $\{20\bar{2}1\}$ , and  $\{22\bar{4}0\}$ . To account for the tetragonality of the  $\gamma$  phase, the crystallographic planes and directions have been expressed according to the rule proposed by Hug *et al.* [15]. The pole figure data was analyzed using the popLA software package [16]. Due to the tetragonality of the  $\gamma$  unit cell, the  $\langle 100 \rangle$  and  $[001]$  reflections are non-equivalent and their peak locations in the  $2\theta$  scans overlap. Similarly, the  $\langle 110 \rangle$  and  $\langle 101 \rangle$  reflections also overlap. To separate the overlapping peaks, the Sample Orientation Distribution (SOD) was computed from a set of measured incomplete pole figures using the WIMV algorithm. Recalculations of the complete pole figures for each overlapping reflection and calculations of the inverse pole figures were then obtained from the SOD. Vickers microhardness measurements were conducted on the DC strip to gauge the variation in mechanical properties through the thickness of the sheet.



## RESULTS

The surface morphology and longitudinal microstructures observed in the DC strip are summarized in Figures 1 and 2. On the wheel side of the cast strip, equiaxed grains composed of fine dendrites were observed (Figure 1a). Comparable microstructures were observed on the free surface of the specimen (Figure 1b) with the appearance of additional dendritic segregation. On both surfaces the dendrite arms were oriented at  $60^\circ$  with respect to each other and had clearly undergone a solid state phase transformation during cooling. Similar dendrite morphologies have been observed in the shrinkage cavities of  $\gamma$  alloys and have been attributed to solidification from a primary hexagonal  $\alpha$  phase [17].

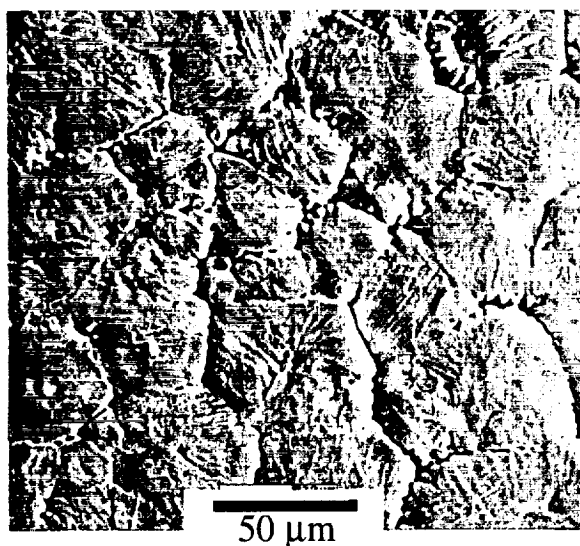
The longitudinal microstructures observed in the DC strip are summarized in Figure 2. The DC strip (Figure 2a) consisted of a nonuniform mixture of massive  $\gamma$  and fine lamellar  $\alpha_2+\gamma$ . The microstructures nearer the wheel side tended to be more equiaxed and columnar while the free side microstructures were more coarse. In localized regions, the columnar or coarse transformed structures continue through the specimen thickness suggesting the occurrence of some directional solidification or nonuniform cooling during solidification. Consistent with prior reports [13], small amounts of porosity have been observed near the wheel surface of the specimen. This porosity has been attributed to solidification shrinkage. After annealing for 48 hrs. at  $1065^\circ\text{C}$  (Figure 2b), the solidification microstructure changed into a nonuniform distribution of equiaxed  $\gamma$  grains ( $\sim 30\text{ }\mu\text{m}$ ), lamellar  $\alpha_2+\gamma$  colonies ( $\sim 30\text{ }\mu\text{m}$ ), and fine  $\alpha_2$  precipitates ( $5\text{ }\mu\text{m}$ ). In addition, it was observed that a thin ( $10\text{-}20\text{ }\mu\text{m}$  thick)  $\alpha$ -case formed on the exterior of the specimen. Though the microstructures appeared to be quite segregated in the DC strip, there was little evidence of chemical segregation. In both the DC and DC+annealed condition, microstructural features were

barely resolvable in the SEM and microhardness remained relatively constant throughout the strip thickness (Figure 3).

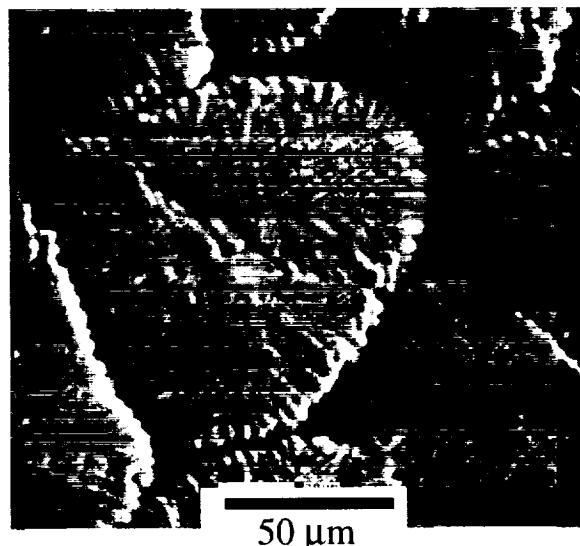
Through thickness texture variations are summarized in the pole figures shown in Figures 4 and 5. In the  $\gamma$ -phase, relatively weak ( $<3 \times \text{random}$ )  $<101]$  type fiber textures were consistently observed in the DC strip with little variance of texture intensity through the strip thickness (Figure 4a). These textures persisted after annealing with virtually no change in texture intensity (Figure 4b). In the  $\alpha_2$ -phase,  $<0002>$ ,  $<10\bar{1}0>$ , and  $<11\bar{2}0>$  fiber textures were all observed in the DC strip (Figure 5a). After annealing, however, this fiber texture was replaced by a weak  $<10\bar{1}0>$  components (Figure 5b).

## DISCUSSION

The microstructures and textures observed in the DC and DC+annealed strips were found to vary considerable through the thickness and along the length. Consistent with prior investigations of rapidly solidified  $\gamma$ -based alloys [18,19], slightly columnar solidification morphologies were observed

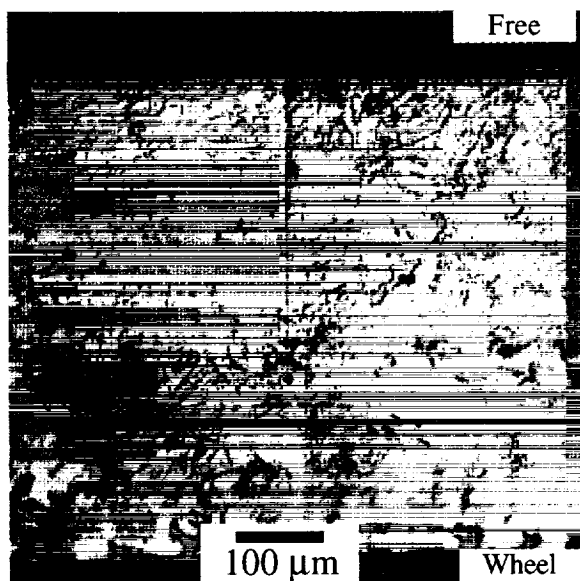


(a)

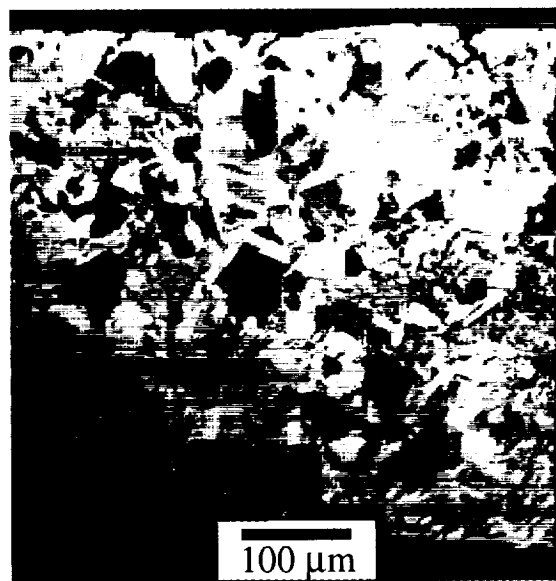


(b)

Figure 1. Backscattered scanning electron micrographs of the surfaces of the DC strip: (a) wheel surface and (b) free surface.



(a)



(b)

Figure 2. Optical micrographs of the DC and DC+annealed strips: (a) DC and (b) DC+annealed. Casting direction is horizontal in both micrographs.

in the DC strips indicating the occurrence of some degree of directional solidification. During cooling the microstructures transformed resulting in more equiaxed features near the wheel surface and more coarse massive and lamellar transformation products near the free surface. Long term anneals at 1065°C caused the solidification structure to break down into a more uniform distribution of  $\gamma$  and  $\alpha_2$  grains and lamellar  $\gamma+\alpha_2$  colonies.

The  $\langle 101 \rangle$  fiber textures observed in the  $\gamma$ -phase textures observed in the DC material are surprising since the primary phase of solidification is hexagonal  $\alpha$ . Typically, solidification textures in chill cast hcp metals/alloys depend on the  $c/a$  ratio [20]. In Mg, for example, where  $c/a$  is slightly less than the ideal value, a  $\langle 11\bar{2}0 \rangle$  fiber texture is observed in the columnar zone whereas in metals such as Zn,

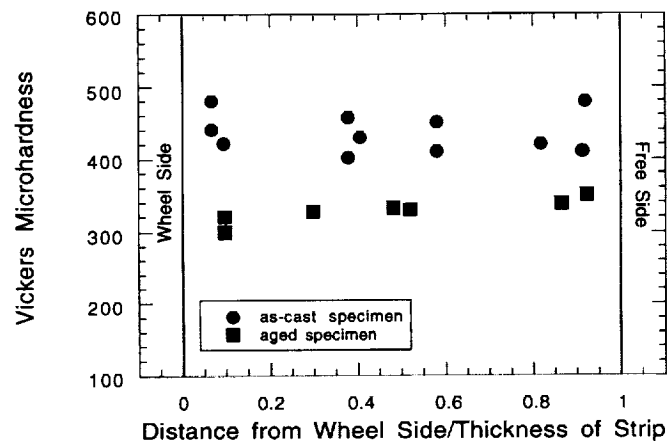


Figure 3. Variation of microhardness through the thickness of the DC strip.

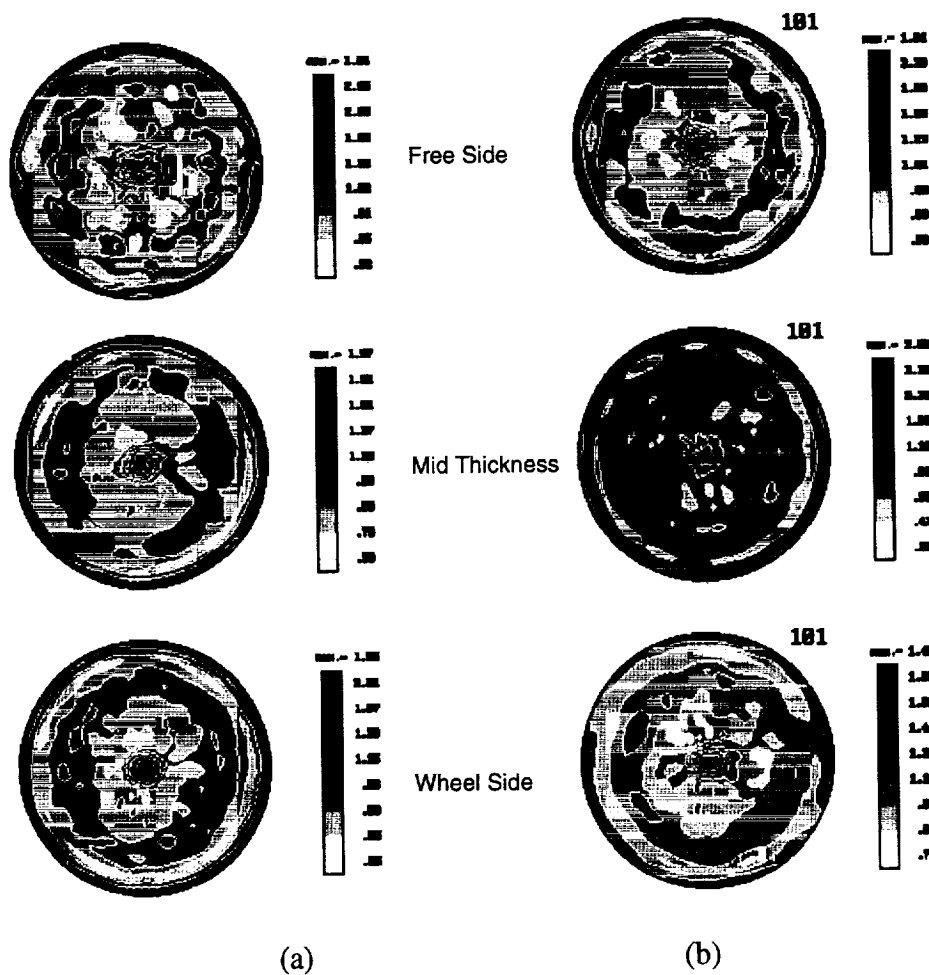


Figure 4.  $\langle 101 \rangle$  pole figures for the  $\gamma$ -phase: (a) DC strip and (b) DC+annealed strip. The pole figures for the annealed specimen were collected from random regions of the strip after the alpha case was ground from the specimen surface.

where  $c/a$  is greater than ideal, a  $\langle 10\bar{1}0 \rangle$  fiber texture is observed. In the chill zone,  $\langle 0001 \rangle$  textures are usually observed (*i.e.*, the basal plane is parallel to the solid/liquid interface). In the case of rapid solidification of hexagonal metals, the basal plane is almost always parallel to the chill contact surface and texture formation seems to match the orientations developed during normal casting [20,21]. These observations have been confirmed in prior investigations of rapidly solidified or twin roll cast  $\gamma$

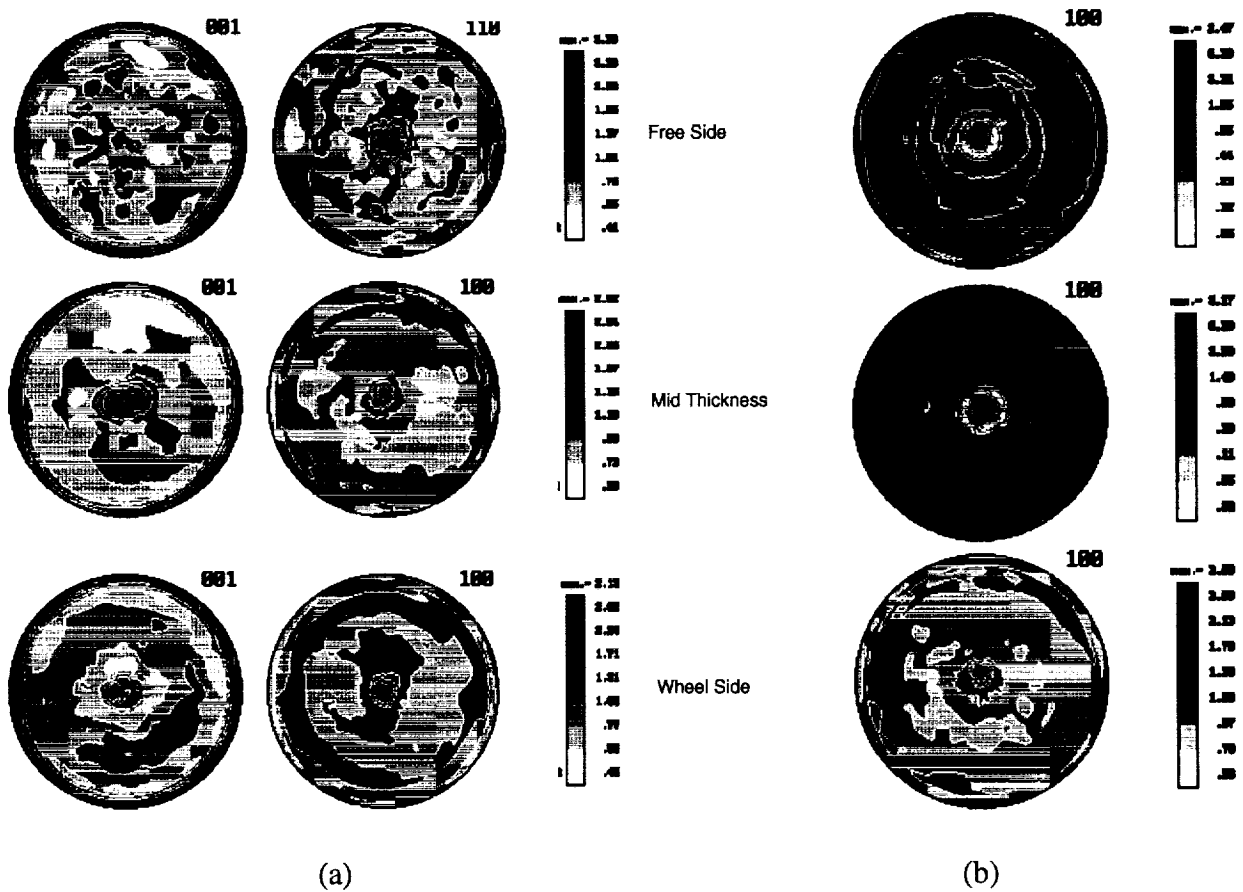


Figure 5. Representative pole figures for the  $\alpha_2$ -phase: (a) DC strip and (b) DC+annealed strip.

The pole figures for the annealed specimen were collected from random regions of the strip after the alpha case was ground from the specimen surface.

sheets. In both cases, a strong  $\langle 111 \rangle_\gamma$  transformation texture with the  $\langle 111 \rangle_\gamma$  axis parallel to the sheet normal  $[2,11]$  while conventionally cast alloys typically exhibit lamellar microstructures with strong  $(\bar{1}1\bar{1})_\gamma$  and  $(0001)_{\alpha_2}$  textures parallel to the direction of heat flow [22]. The observed textures are more reminiscent of deformation textures. For example, hot working below the  $\alpha$ -

transus or hot pack rolling results in recrystallized fine-grained microstructures with  $\{101\}_\gamma$  textures [23] or cube textures [24] respectively where the c-axis aligns in the transverse direction [10,23,25]. It is speculated that these textures are a result of the MORST process. The microstructural observations suggest that nonuniform cooling results as the material is cast and as the strip piles up on the bottom of the collection chamber. Some lattice rotation is expected as the liquid metal strikes the chilled copper wheel. In addition, assuming that the top layers of the DC strip cool slower than the side that was in contact with the water-cooled walls of the vessel or the strip that was not contacting any solid surface, it is expected that the material nearer the wheel side will experience a slight compressive stress due to the accumulation of material on top of it and the residual stresses caused by nonuniform cooling. The  $\langle 10\bar{1}0 \rangle$  textures observed in the  $\alpha_2$ -phase after annealing can be described as an artifact of the solidification texture. In a prior investigation of forged  $\gamma$ -based alloys [5], it was shown that recrystallized grains of  $\gamma$  and  $\alpha_2$  can exhibit the orientation relationship  $(1\bar{1}\bar{1})//(\bar{0}001)$  and  $\langle 110 \rangle//[10\bar{1}0]$  as opposed to the Blackburn relationship typically observed in lamellar structures. Further investigations of texture development in DC and DC+A  $\gamma$ -strips are in order to properly elucidate the mechanisms of texture formation.

## SUMMARY AND CONCLUSIONS

Texture analysis of DC  $\gamma$ -TiAl strips indicated that  $\langle 101 \rangle$  deformation textures persisted in the  $\gamma$ -phase while  $\langle 0002 \rangle$ ,  $\langle 10\bar{1}0 \rangle$ , and  $\langle 11\bar{2}0 \rangle$  texture components were observed in the  $\alpha_2$ -phase. These textures are the result of nonuniform cooling which results in lattice rotation and residual stresses.

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# **CHAPTER III**

## **MICROSTRUCTURAL AND MECHANICAL PROPERTIES EVALUATIONS OF TITANIUM FOILS PROCESSED VIA THE MELT OVERFLOW PROCESS**

### **INTRODUCTION**

The processing of titanium foils by conventional ingot metallurgy (IM) techniques involves casting ingots, hot forging into billets, followed by several hot rolling, heat treatment, and surface grinding sequences to produce plate or strip that is suitable for cold rolling to foil gauge. Using these techniques, processing losses exceeding 50% are not uncommon making commercial production of titanium foils very expensive. Recently, Gaspar *et al.* [1-3] have reported success in direct casting sheets of conventional titanium alloys and titanium-based ordered intermetallic compounds using a single-chill-roll casting technique called melt overflow rapid solidification technology (MORST). Using this technique, near-net-shape foils have been continuously cast into  $\sim 500 \mu\text{m}$  thick  $\times 10$  cm wide  $\times 3$  m long sheets which were successfully ground, cold rolled, or hot pack rolled to foil gauge ( $\leq 100 \mu\text{m}$  thickness). In comparison to IM processing techniques, the potential advantages of foil production from direct cast (DC) strips are improved purity, increased chemical homogeneity, and a reduction in processing losses resulting in lower processing costs. While the microstructures, mechanical properties, and textures of IM titanium alloys have been extensively characterized, they have not yet been addressed for DC titanium. In the present work, the microstructures, mechanical properties, and crystallographic textures developed in DC strips and in cold rolled foils produced from DC strips are compared with those of IM titanium foils.

## Experimental Procedure

Titanium strips were cast in the plasma melt overflow furnace at Ribbon Technology Incorporated, Columbus, Ohio. The plasma melt overflow furnace combines plasma arc melting in a cold copper hearth with MORST by rotating the cold copper hearth about the same axis of rotation as the chill roll to overflow liquid onto the circumference of the chill roll [1-3]. The chemical compositions of the DC titanium strip/foil and of a conventional ingot metallurgy (IM) foil supplied for comparison purposes are given in Table 1. The DC material was prepared from conventional purity (CP) titanium [2]. However, during processing of the DC strip used in this investigation, approximately 20% Ti-6Al-4V scrap was accidentally mixed with the CP-Ti scrap. This addition was not discovered until the alloy strip had been cast, cold rolled and annealed (CR).

Portions of the DC strip were supplied to Texas Instruments, Materials and Controls Division, Attleboro, MA for cold rolling. Cold rolling was accomplished in two rolling and annealing steps where the strip was initially cold rolled to approximately 50% of its original thickness followed by vacuum annealing at 700°C for two hours. The strip was then rolled to a final thickness of 0.17 mm and again vacuum annealed at 700°C for two hours. The IM sheet (0.15 mm thickness) was processed using conventional techniques.

**Table 1. Chemical compositions of titanium strips/foils investigated**

Processing Method	Wt. %				Wt. ppm			
	Ti	Al	V	Fe	O	N	H	C
DC	balance	1.29	0.93	0.19	3600	450	8	150
IM	balance	0.043	0.008	0.12	1960	130	57	380

DC = direct cast, IM = ingot metallurgy

For texture analysis, portions of each alloy strip were cut into small pieces, mechanically polished, and etched with Kroll's reagent to remove any residual deformation layers. Texture variations were measured using the x-ray diffraction technique on a Philips X'Pert PW3040 MRD x-ray diffractometer operating at 40 kV and 45 mA. The following incomplete pole figures were measured using Ni filtered  $\text{CuK}\alpha$  radiation to determine textures in the  $\alpha$  phase:  $\{0002\}$ ,  $\{10\bar{1}0\}$ ,  $\{10\bar{1}1\}$ ,  $\{11\bar{2}0\}$ , and  $\{11\bar{2}2\}$ . The pole figure data was analyzed using the popLA software package [4].

Dog-bone shaped tensile specimens 45 mm long with a gage section  $11.4 \text{ mm} \times 6.4 \text{ mm}$  were machined from the cold rolled foils. Multiple specimens were machined parallel to and perpendicular to the rolling direction. Tensile tests were performed at room temperature on a computer interactive ATS Model 1630 universal testing machine operated at constant crosshead velocities corresponding to an initial strain rate of  $7.4 \times 10^{-3} \text{ s}^{-1}$ .

## Results and Discussion

### Microstructure

In the DC strip, equiaxed grains with grain diameters approaching 35  $\mu\text{m}$  were observed. During solid-state cooling, the alloy transformed martensitically to a mixture of acicular  $\alpha$  and retained  $\beta$  phases. A typical optical microstructure is shown in Figure 1. In general, the strip was fully dense and contained no visible porosity or cracks. After cold rolling and annealing, microstructures consisting of fine equiaxed  $\alpha$  grains were observed in both the DC and IM foils. Nominal grain diameters were 18  $\mu\text{m}$  in the DC foil and 14  $\mu\text{m}$  in the IM foil (Figure 2).

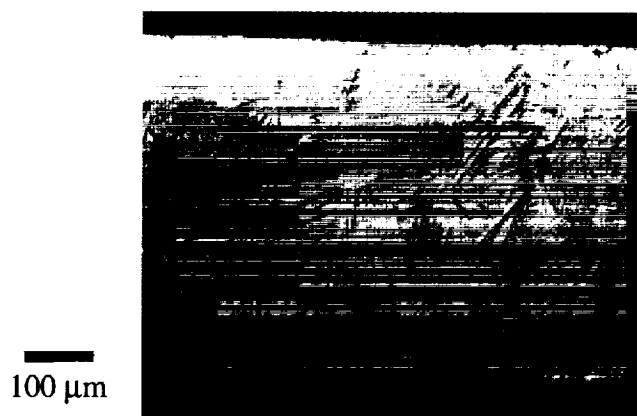


Figure 1. Optical micrograph of DC titanium strip. Casting direction is horizontal.

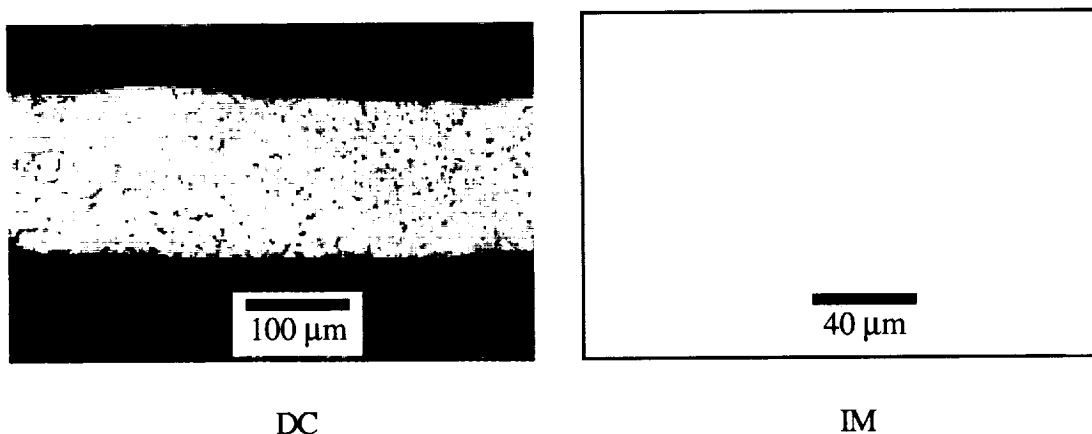


Figure 2. Optical micrographs of DC and IM titanium foils used in this study. The rolling direction is horizontal

### Texture

Texture can be described as a non-random distribution of grain orientations that occurs during the manufacture of materials. Significantly different textures can result from solidification, deformation, recrystallization, and phase transformations and may lead to anisotropic mechanical properties. The experimental textures for the DC strip and for the DC+CR and IM+CR foils are represented in the pole figures displayed in Figures 3 and 4. In the DC strip, relatively weak ( $\sim 1.8 \times$  random) fiber textures were observed with the major poles oriented parallel to the strip normal in the  $\{11\bar{2}0\}$  direction with some components nearly parallel to the casting and transverse directions in the  $\{0002\}$  pole figure. The type of textures corresponding to those where the basal plane is parallel to either the chill or wheel surface of the specimen are typical in rapidly solidified hcp metals produced via conventional chill casting or melt spinning [5,6].

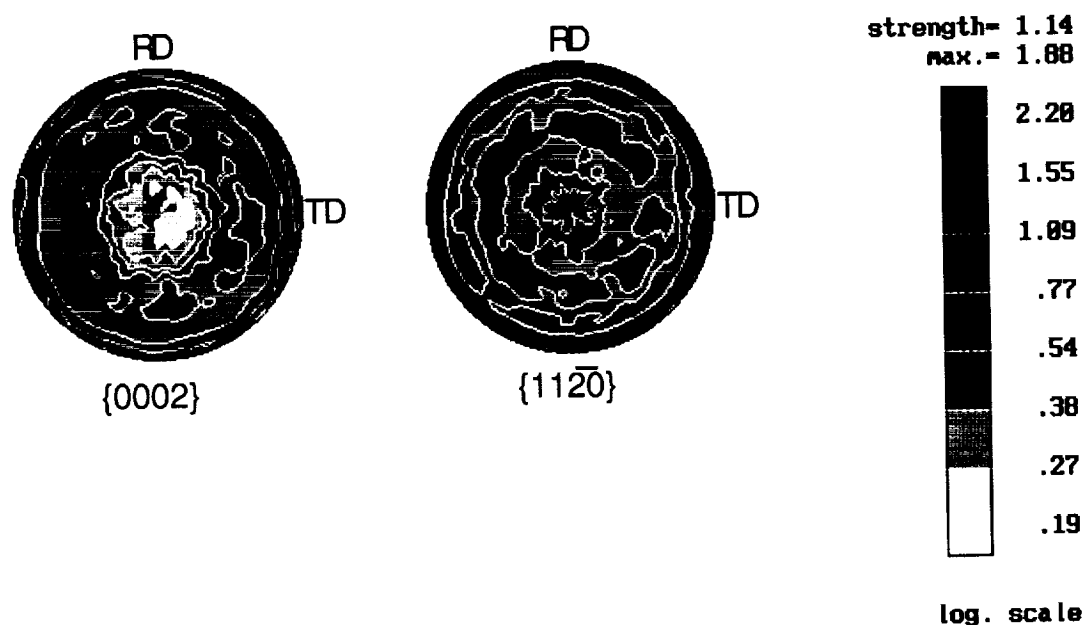


Figure 3.  $\{0002\}$  and  $\{11\bar{2}0\}$  indirect pole figures for DC titanium strip. The casting direction is labelled RD on the pole figures.

The post CR textures for DC and IM foils are shown in Figure 4. After CR, the texture intensifies ( $\sim 3 \times$  random). In both foils, the c-axes were concentrated in the normal direction (ND)-transverse direction (TD) plane tilted approximately  $35^\circ$  from the ND towards the TD. Such split textures are commonly observed in IM titanium foils produced by cold rolling [7-10].



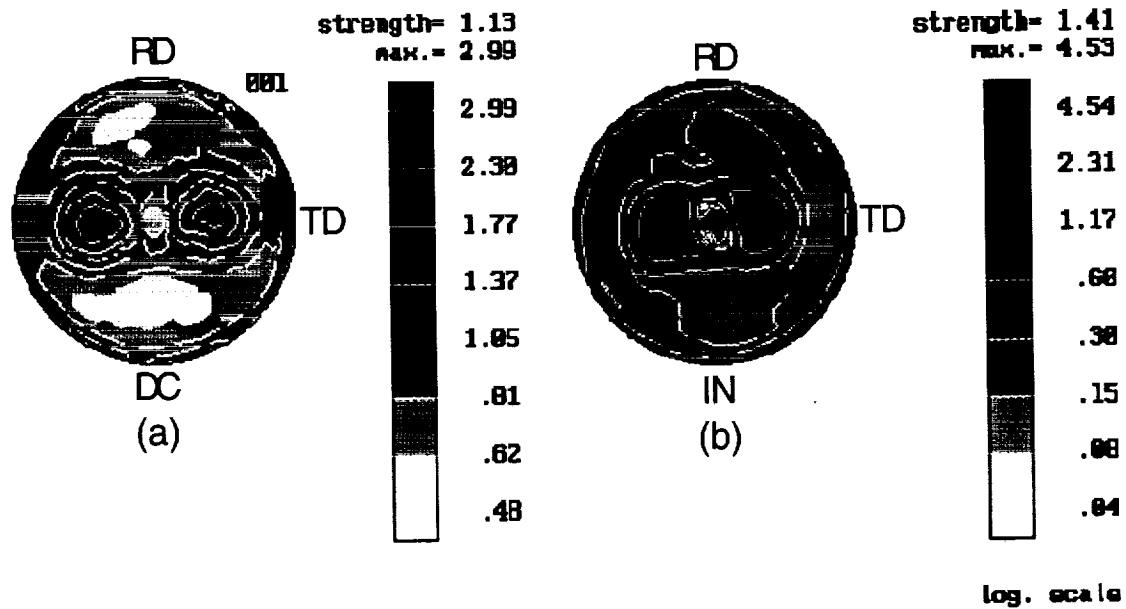


Figure 4. {0002} indirect pole figures for DC and IM foils.

### Mechanical Properties

Mechanical properties results are presented in Table 2 along with the results obtained in a recent investigation of DC titanium alloys [2]. It is difficult to make an accurate comparison of mechanical properties due to the differences in composition between the DC and IM foils, however, some general observations can be noted. In agreement with Gaspar *et al.* [2], the room temperature yield stress (YS) and ultimate tensile stress (UTS) were relatively anisotropic in the IM titanium specimen. It was additionally observed that the IM specimens exhibited ~40% greater tensile elongation's to failure than the DC specimens. Consistent with the observations of Gaspar *et al.* [2], YS and UTS were relatively anisotropic in the DC foils. The elongation, however, was noticeably lower in the transverse direction. They attributed this difference to crystalline anisotropy resulting from unidirectional rolling.

## **Summary and Conclusions**

In general, this study has confirmed prior reports that high quality DC foils with mechanical properties comparable to IM foils can be successfully produced using the MORST process. Furthermore, these foils exhibit the same microstructures and texture components (with a larger texture component in the case of DC foils) as IM processed foils with lower interstitial impurity contents.

**Table 2. Tensile Properties of CP-Ti Foils**

Sample [reference]	Orientation	YS (MPa)	UTS (MPa)	$\epsilon_p$ (%)
IM [this study]	L	545±7	645±7	16.3±2.5
IM [this study]	T	515±35	620±28	16.8±1.1
IM CP-Ti [2]	L	339	459	31.5
IM CP-Ti [2]	T	362	438	28.7
DC [this study]	L	910	1010	15.1
DC [this study]	T	978±68	1170±17	7.3±1.4
DC Ti-1.25Al-0.8V [2]	L	762	841	18.6
DC Ti-1.25Al-0.8V [2]	T	807	887	7.4

L = longitudinal, T = transverse, IM = ingot metallurgy, DC = direct cast, base strain rate =  $7.4 \times 10^{-3} \text{ s}^{-1}$

NOTE: in reference [2], a nominal strain rate of  $10^{-4} \text{ s}^{-1}$  was used

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# **CHAPTER IV**

## **TEXTURE AND MICROSTRUCTURE OF ALPHA-2 TITANIUM SHEET PRODUCED BY PLASMA CAST OVERFLOW PROCESS**

### **Introduction**

The production of high temperature materials at a lower cost having equal or improved properties is an ongoing materials concern. Studies are primarily centered on aluminides of nickel, iron, and titanium (1). Development of advanced materials for high temperature applications in aerospace, automotive, and other industries are among those systems receiving the greatest attention (3). Titanium aluminides have been the focus of interest for lightweight, high-temperature applications because of their low density. Production of these materials is most commonly performed using foil-fiber-foil (FFF) fabrication method (2). There are still several problems associated with the reproducibility of manufacturing these foils. A new technique was developed by Ribbon Technology (Ribtech) for the production of thin sheet metals to be used for future fabrication of metal matrix composite materials. The process involves Rapid Solidification Melt Overflow Technique (MORST) that results in considerable cost reduction and eliminates the hot working steps during production reducing lead times (3-4). The objective of the present work is to characterize the microstructure and crystallographic texture of alpha-2 (Ti-24Al-11Nb) as-cast and heat-treated at 900°C and 1000°C and cold rolled produced via MORST process.

## **Experimental Procedure**

The as-cast alpha-2 materials were fabricated at Ribtech via Rapid Solidification Melt Overflow Process. Samples of the as-cast alpha-2 material were cut and epoxy mounted for texture and microstructural analysis. Each sample was polished to 0.05 $\mu$ m Al<sub>2</sub>O<sub>3</sub> to produce a flat & stress free surface ideal for texture and microstructural analysis. Microscopy analysis was performed using an Environmental Scanning Electron Microscope (ESEM).

After the initial texture and microstructural analysis of the as-cast material, samples were heat treated at 900°C and 1000°C, for thirty minutes in argon gas. The materials were then cold-rolled until failure and at each step of the process, a complete microstructural analysis was performed on the polished samples.

The samples were etched using Kroll's Reagent solution (1-3mL HF, 2-6mL HNO<sub>3</sub>, and 100mL H<sub>2</sub>O) for microscopy analysis. The microstructure was first examined using an optical microscope. The ESEM was then utilized to examine the finer microstructure. X-ray microanalysis was performed using Electron Dispersive Spectroscopy (EDS) in ESEM to investigate the difference in the chemical composition of the material after processing.

Texture was analyzed via the X-ray Diffractometer for samples at all stages of processing. Using Popla software, orientation distribution functions (ODF's) were calculated by Roe's approach to spherical harmonic method. The pole figures were recalculated using the ODF's. The planes used to calculate the pole figures were as follows:  $\langle 110 \rangle$ ,  $\langle 002 \rangle$ ,  $\langle 112 \rangle$ ,  $\langle 200 \rangle$ , and  $\langle 202 \rangle$ . Only the  $\phi = 0, 20, 30, 40$  angles were used from the pole figure calculations for texture analysis.

## **Results and Discussion**

The microstructure and texture of the as-cast material was compared to the results from previous studies (5,8). Preliminary texture analysis revealed random texture in the as-cast material. Preliminary microstructural analysis showed that the as-cast material exhibited a Widmanstatten structure (basketweave). Past studies by Ribtech have also shown the microstructure of as-cast alpha-2 titanium aluminide exhibiting Widmanstatten structure (5). Heat treatment of the pack-rolled foils resulted in the same microstructure as the as-cast material; however, small pores were observed scattered randomly throughout the microstructure. Pack rolling the as-cast strip eliminated the internal porosity as well as the surface roughness during the standard foil grinding operation (5) as shown in Fig. 1. The features are not as pronounced as a result of the rapid cooling process of the MORST technique (5).



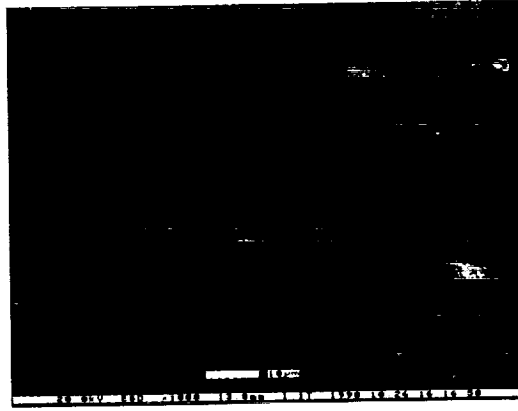
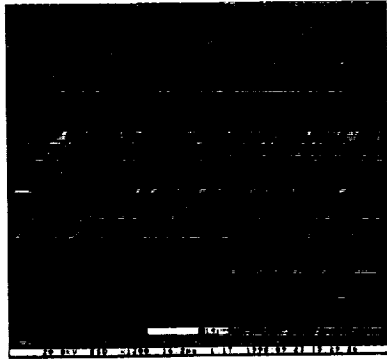


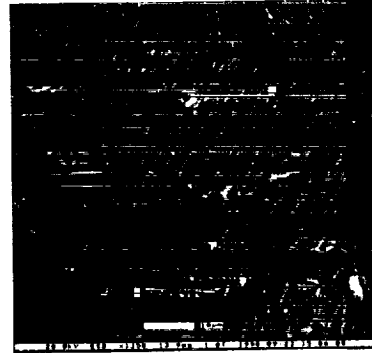
Fig 1 Initial as-cast microstructure

Heat-treatment at  $900^{\circ}\text{C}$ , resulted in slight surface oxidation, which becomes apparent through surface discoloration (yellowish). The heat-treated material still exhibited a Widmanstatten microstructure. Heat-treatment at  $1000^{\circ}\text{C}$ , resulted in slight surface oxidation, which becomes apparent through surface discoloration (rainbow). These results suggest that less oxidation occurred for the material annealed at  $1000^{\circ}\text{C}$  compared to that at  $900^{\circ}\text{C}$ . The samples were then rolled using a manual-rolling machine. It was expected that heat treatment at  $1000^{\circ}\text{C}$  would result in more ductility, but the results showed less ductility. It was possible to deform rolled samples heat-treated at  $900^{\circ}\text{C}$  by 40%, whereas heat treatment at  $1000^{\circ}\text{C}$  reduced only 24% before fracture. Possible explanations for the decrease in ductility at the higher temperature can be based on the quenching process or phase transformation. Quenching can produce internal stresses, which can cause decrease in material strength. This may occur due to incompatibility between phases. Due to the range of temperatures involved with heat

treatment process, deformations due to phase transformation may be the other cause for internal stresses. Titanium alloy (Ti-24Al-11Nb) at 900°C has a two phase  $\alpha_2 + \beta$  phase but, at 1000°C it goes through a phase transformation yielding  $\alpha_2 + B_2$  (ordered b phase) (8). The transformation of  $\beta$  to ordered b phase might explain the reduction in ductility observed for these materials. The samples were investigated further by performing an EDS analysis at all steps of the process. The results showed no significant difference in chemical composition in any of the samples. The microstructure at all stages of the process is shown in FIG. 2 (a-b) and FIG. 3 (a-b). FIG. 3a suggests slight recrystallization (grain growth) of a selected number of grains.

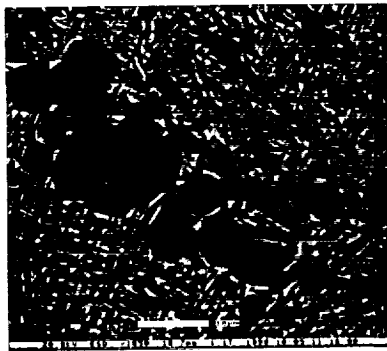


(a)



(b)

Fig 2 (a) Annealed @ 900°C for .5h (b) Annealed @ 900°C for .5h and cold rolled



(a)



(b)

Fig 3 (a) Annealed @ 1000°C for .5h (b) Annealed @ 1000°C for .5h and cold rolled

X-ray Texture analysis was performed on as-cast and processed samples of the alpha-2 material using a Phillips X'pert PW3040 MRD X-ray Diffractometer operating at 40 kv and 50 mA. The results of the X-ray Diffractometer  $2\Theta$  scans are shown in Fig 4. The as cast texture for  $\phi = 20^\circ\text{C}$  is shown in Fig. 5. ODF's were analyzed at orientations of  $\phi = 20^\circ\text{C}$  to compare the results of as-cast alpha-2 to its heat-treated condition. The as-cast alpha-2 exhibited texture in the  $\phi = 20$  plane  $(-3121)$ . Heat-treatment at both  $900^\circ\text{C}$  and  $1000^\circ\text{C}$  resulted in 3x random texture intensity in  $(-3121)[t\ u\ v\ w]$  and  $(-3123)[t\ u\ v\ w]$ . Rolling for  $900^\circ\text{C}$  heat treatment condition reduced the texture intensity returning similar results to the as-cast texture but, the  $1000^\circ\text{C}$  condition seemed to remain unchanged (see Figs 6 and 7). All of the results are summarized in Table 1.

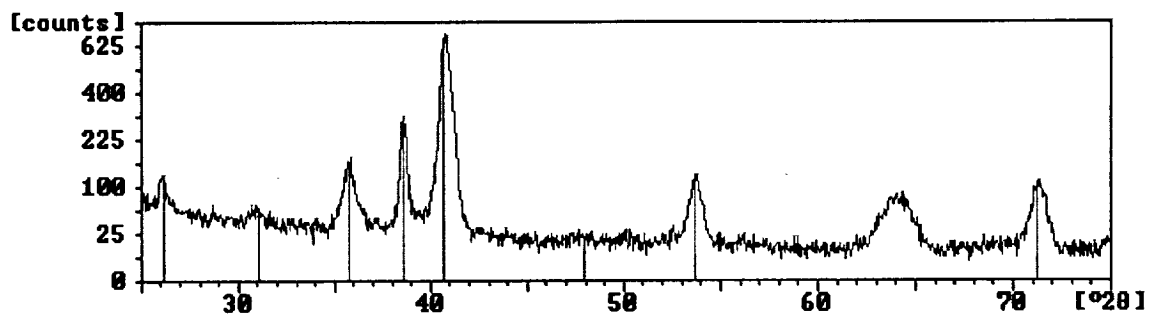


Fig 4 2-Theta Scan for As-cast Ti-24Al-11Nb

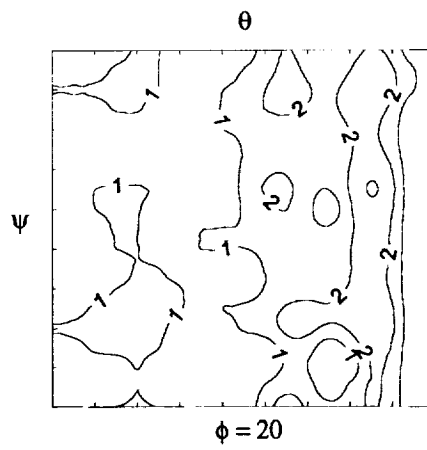


Fig 5 Initial as-cast alpha-2 texture

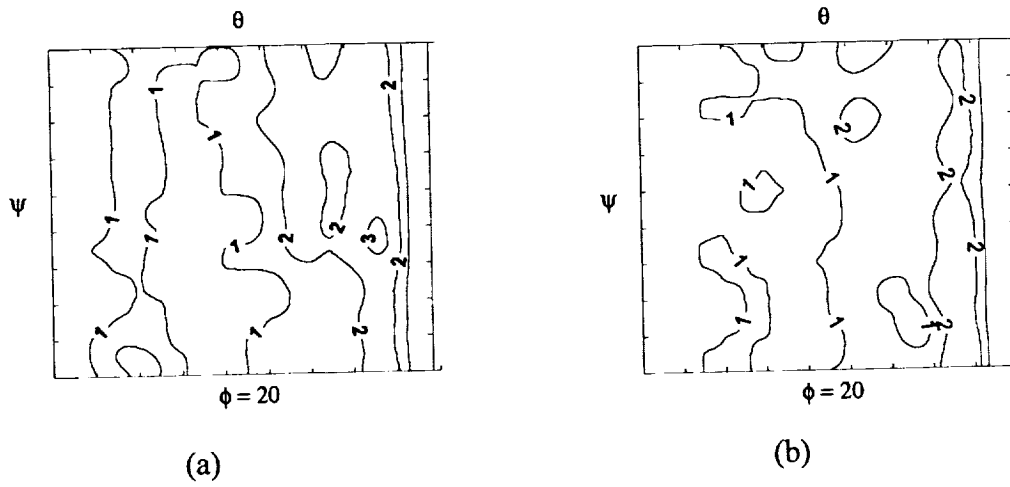


Fig 6 Alpha-2 (a) annealed @ 900°C for .5h (b) annealed @ 900°C for .5h and cold rolled

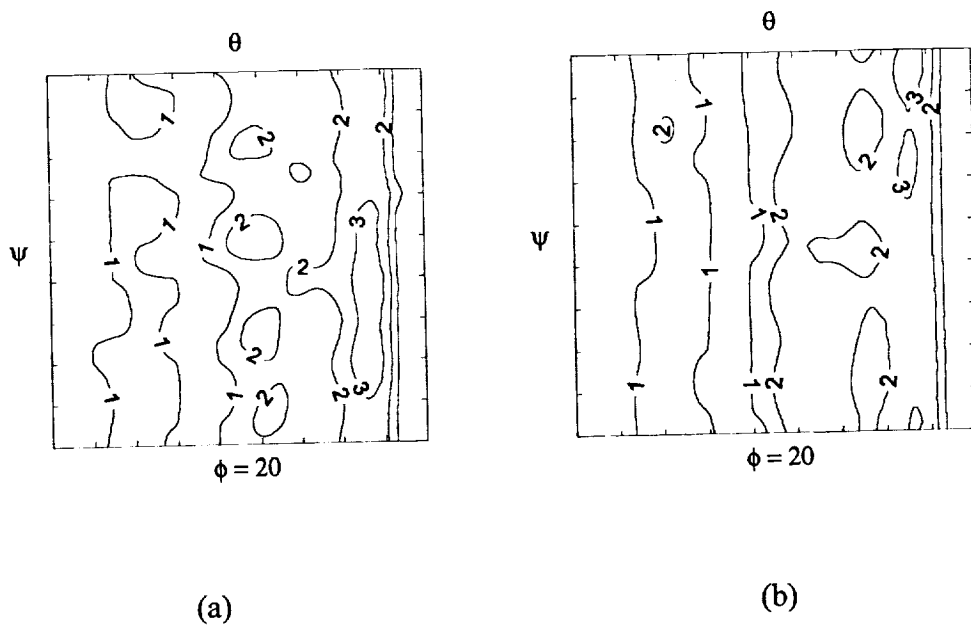


FIG 7 Alpha-2 (a) annealed @ 1000°C for .5h (b) annealed @ 1000°C for .5h and cold rolled

## Summary

In summary, processing alpha-2 via MORST results in fast cooling which leads to the Widmanstätten microstructure and weak texture exhibited by the structure of the as-cast material. The characterization of the post processing (heat treatment & cold rolling) of alpha-2 titanium foil suggests two distinct phenomena at the two heat treatment temperatures of 900°C and 1000°C. The microstructure of the material annealed at 900°C remains unchanged while annealing at 1000°C results a phase change from  $\alpha_2 + \beta$  to  $\alpha_2 + \text{B2}$  phase. Partial recrystallization was also observed at 1000°C. A ductility of 24% and 37% was measured for the 1000°C heat treatment and 900°C heat treatment procedures. Annealing also intensified the texture of Ti-24Al-11Nb.

**Table 1: Summary of characterization of Ti-24Al-11Nb**

(times random)

	AS-CAST	HEAT TREATED		ROLLED	
		900°C	1000°C	900°C	1000°C
Microstructure	Widmanstätten	Recovery	Start of recrystallization		
(2 0 2 1) $\phi = 0^\circ$	1	2	2	1	1
(3 0 3 1) $\phi = 0^\circ$	2	2	2	1	1
(-3 1 2 1) $\phi = 20^\circ$	2	3	3	2	3
(-3 1 2 3) $\phi = 20^\circ$	1	1	2	1	1
(-2 1 1 0) $\phi = 30^\circ$	1	2	2	1	1
(-3 2 1 0) $\phi = 40^\circ$	2	2	2	1	1
(3 2 1 3) $\phi = 40^\circ$	2	2	2	1	1
Reduction				37%	24%



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